FINAL WORKPLAN
SAMPLING AND ANALYSIS OF PROPERTIES IN THE VICINITY OF THE EXIDE FACILITY (VERNON, CALIFORNIA)

Prepared for

Department of Toxic Substances Control
8800 Cal Center Dr
Sacramento, CA 95826

Prepared by

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November 18, 2015

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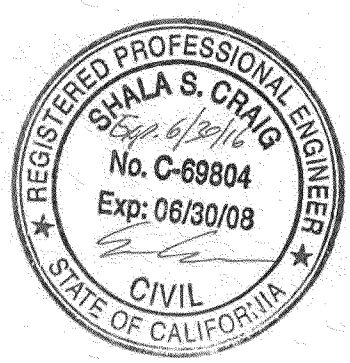

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ACRONYMS AND ABBREVIATIONS

AL	Action Level
Cal-EPA	California Environmental Protection Agency
CCR	California Code of Regulations
CFR	Code of Federal Regulations
COC	chain-of-custody
CDPH	State of California Health and Human Services Agency, Department of Public Health
DQOs	Data Quality Objectives
DL	detection limit
DTSC	Department of Toxic Substances Control
ELAP	Environmental Laboratory Accreditation Program
EPA	Environmental Protection Agency
ft	feet
ft^2	square feet
HUD	Department of Housing and Urban Development
IMWP	Interim Measures Workplan
LAC	Los Angeles County
LBP	lead-based paint
mg/kg	milligrams per kilogram
mg/cm^2	milligrams per square centimeter
NIST	National Institute of Standards and Technology
OEHHA	Office of Environmental Health Hazard Assessment
OSHA	Occupational Safety and Health Administration
POC	point of contact
PSHEP	Project Safety Health and Environmental Plan
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
SI	Site Investigation
SOW	scope of work
SCAQMD	South Coast Air Quality Management District
USA	Underground Services Alert
Workplan	Site Characterization Workplan
XRF	X-ray fluorescence

1 INTRODUCTION AND BACKGROUND

1.1 Introduction

On October 29, 2015, Parsons was tasked by the Department of Toxic Substances Control (DTSC) with the preparation and implementation of a Workplan addressing sampling and analysis at 1,000 residential and sensitive-use properties located near the former Exide Technologies (Exide) battery recycling facility in Vernon, California. Lead emissions from the former Exide facility are suspected of affecting surface and near-surface soils in surrounding areas as a result of aerial deposition. A number of previous investigations have been performed to characterize soil impacts at various properties near the Exide site. DTSC's preliminary evaluation of the soil sampling results collected to date at the Exide facility suggests that the geographic distribution of Exide's lead emissions may extend 4,500 feet to 9,000 feet (ft) north and south into portions of Maywood, Boyle Heights, East Los Angeles, City of Commerce, Bell, and Huntington Park (Preliminary Investigation Area), as shown in Figure 1. As a result, DTSC has contracted Parsons to determine if aerially deposited lead may have affected off-site residential soils within the Preliminary Investigation Area at concentrations of potential concern from a human health perspective. The DTSC is developing criteria for prioritizing cleanup of the off-site residential soils.

The goal of this investigation is to identify those residential properties that contain lead soil concentrations equal to or greater than 1,000 mg/kg at hazardous levels of lead. Properties with these lead concentrations in soil are considered having the greatest lead exposure potential. After these properties are identified, an Interim Measures Workplan (IMWP) will be prepared that describes procedures for removing affected soil and performing site restoration work at those properties. Once the properties with elevated levels of lead are identified, cleanup will be implemented in accordance with the IMWP. The criteria used to prioritize soil removal at sampled properties with lead concentrations less than 1,000 mg/kg will be further described in the IMWP following consultation with the DTSC and the local community.

The Workplan is organized as follows: Section 1 presents an introduction, background and scope of work (SOW). Section 2 presents the pre-investigation activities. Section 3 presents the planned field sampling and data collection activities. Section 4 presents the reporting structure. Section 5 presents references cited in this Workplan.

1.2 Background

The former Exide Facility is located at 2700 South Indiana Street in the City of Vernon, California (Figure 1). This industrial property occupies approximately 15 acres, bounded by South Indian Street to the west, 26th Street to the north, Bandini Boulevard (Bandini) to the south, and industrial properties to the east. The facility was formerly used for lead battery recycling. The immediate surrounding area is industrial.

To determine whether off-site residential soils had concentrations of selected constituents that were greater than background or residential screening levels, Exide's contractors, Advanced GeoServices Corp. and ENVIRON International Corporation, conducted soil sampling at residential properties and two schools near the Site in November 2013. Additional soil samples were collected from a background area approximately 14 miles to the south of the facility.

Air dispersion modeling based on the South Coast Air Quality Management District (SCAQMD) requirements identified a preliminary indication of the area in which Exide emissions may have resulted in lead-impacted soil near the Site. Based on this air modeling, soil sampling took place in two residential areas that were identified as having the greatest potential for elevated lead impacts. The Northern Assessment Area for soil sampling is located in Boyle Heights and East Los Angeles; the Southern Assessment Area is located in Maywood.

Nineteen properties were sampled in the Northern Assessment Area, and twenty properties were sampled in the Southern Assessment Area. The soil sampling results were compared to the background results and to California Environmental Protection Agency (Cal-EPA) Office of Environmental Health Hazard Assessment (OEHHA) health screening levels.

Soil lead concentrations exceeding the OEHHA residential soil screening value of 80 mg/kg were identified in both the Northern and Southern Assessment Areas. No attempt was made to attribute observed lead concentrations to specific sources, although it is recognized that, due to the heavily industrialized and densely populated nature of the area, multiple sources exist, including Exide's historic emissions. Other potential lead sources that have affected the soils in the Study Area include deposition from leaded fuel combustion emissions (e.g., from gasoline combustion prior to lead phase-out) and from lead-based paint that is present on virtually most structures in these areas.

Based on the review of the initial soil sampling results and the results of more detailed subsequent sampling, as many as 10,000 properties in the Preliminary Area of Investigation have been identified by the DTSC as properties that may have been impacted by the Exide facility's past emissions.

The following SOW is addressed in this Workplan and will be implemented at each of the first 1,000 residential properties as part of this investigation:

1. Conduct soil sample screening on each property at up to 15 locations on lawn areas, bare soils, garden areas, play areas, and roof drip-zones using an X-ray fluorescence (XRF) analyzer; two of the XRF samples representing the two largest sampling areas will be submitted to a fixed laboratory for confirmatory analysis.
2. Conduct lead-based paint (LBP) screening on each property using an XRF analyzer at up to six exterior structure locations. Paint chip samples will be collected from the main dwelling and from any additional dwellings and structures, if access agreement for the property allows collecting chipped pieces of paint from the surface of the exterior of buildings.

2 PRE-INVESTIGATION ACTIVITIES

2.1 Health and Safety

Parsons and its subcontractors are responsible for operating in accordance with the most current requirements of Title 8, California Code of Regulations (CCR) Section 5192 (8 CCR 5192); and Title 29, Code of Federal Regulations (CFR) Section 1910.120 (29 CFR 1910.120), Standards for Hazardous Waste Operations and Emergency Response. Onsite personnel are responsible for operating in accordance with all applicable regulations of the Occupational Safety and Health Administration (OSHA) outlined in 8 CCR General Industry and Construction Safety Orders; 29 CFR 1910; and 29 CFR 1926, Construction Industry Standards; and with other applicable federal, state, and local laws and regulations. All personnel must operate in compliance with all California OSHA requirements.

A project-specific health and safety plan (Parsons, 2015a) has been prepared in compliance with above regulations and DTSC health and safety requirements. As minimum safety requirements for the work, all subcontractors must evaluate job hazards analyses, prepare a site-specific subcontractor health and safety plan , and review and accept the Parsons Project Safety Health and Environmental Plan (PSHEP). The field superintendent and the project managers are authorized to issue a stop work order at any time if deemed necessary due to safety concerns. Each site worker will attend a detailed project orientation on the first day work and all workers will attend daily tailgate meetings. Activity hazards analysis will be reviewed daily at the tailgate meetings in order to inform each employee of potential hazards associated to each job step (e.g. exposure to site contaminants, biological hazards, traffic, etc.). Due to the low risk nature of the scope of work, job tasks are anticipated to be conducted in Level D PPE.

Particular attention will be given to minimizing impacts to the residents and their surrounding neighbors. This will include establishing clear work zones and areas where the public may not enter.

Chemical exposure to lead in soil for site workers is anticipated to be of low risk for this project. There is no dust generation as part of the sampling activities as soil disturbance is very low. As such exposure due to inhalation is not of concern. Exposure due to ingestion may pose a risk, which can be easily mitigated by proper use of Level D PPE. Hands and shoes may come in direct contact with potentially contaminated soil. Therefore, workers will be required to wear steel toed work boots, latex gloves, high visibility vests, and hard hats as part of their Level D PPE. Handling of soil, soil samples, and sampling equipment is only allowed while wearing latex gloves, or work gloves over latex gloves. After sampling activity is completed, the latex gloves will be discarded and hand washing will be required. Additionally, to prevent track-out off-site, work boots will be decontaminated by brushing off any loose soil on site, and washing the boots with water.

2.2 Regulatory Clearances

The sampling activities will be conducted within private residences; therefore, no permit requirements are necessary with the local jurisdictions. If necessary, encroachment permits will be obtained from the local municipality if equipment will be present within public rights-of-way and “No Parking” areas must be established.

2.3 Project Team

Due to the number of stakeholders on this public project, compliance with the chain of command and lines of communication is an absolute necessity for proper implementation of the Workplan. The following subsections list the authority points of contact (POCs) to be considered during the course of work.

The site investigation (SI) will be collectively managed by the DTSC. The nature of each party's responsibilities is discussed below.

2.3.1 DTSC Contract Management Representative

Mr. Raymond Leclerc, PE, of the DTSC is responsible for overall coordination and organization of the Exide project, including this investigation work. He can be reached at (916) 255-3528. Ray may delegate authority to DTSC field representative for field-related decisions.

2.3.2 DTSC Project Manger

Mr. Peter Ruttan, will represent the DTSC. He will review and approve the Workplan and will coordinate all environmental activities with Parsons. He can be reached at (916) 255-3630.

2.3.3 Parsons

Ms. Shala Craig, PE is Parsons' Project Manager for providing environmental services to the Design Team. In this capacity, she will be the primary liaison between the DTSC and Parsons. She can be reached at (310) 612-3393. Mr. Tom Blaney is Parsons' Field Operations Director and will be responsible for all field work coordination. He can be reached at (626) 440-6067.

3 FIELD INVESTIGATION ACTIVITIES

The field investigation methods are designed to meet the overall objectives of the SOW as described in Section 1.3. The sampling strategy, field and laboratory methodologies, and quality assurance/quality control (QA/QC) measures to provide data of sufficient quantity and quality are described in this section. A Quality Assurance Project Plan (QAPP) and Data Quality Objectives (DQOs) have also been developed by Parsons. The purpose of the QAPP is to present the organization, objectives, functional activities, and specific QA/QC activities in support the proposed sampling. The QAPP and DQOs are provided in Appendix A.

3.1 Property Access

All property access agreements will be handled by the DTSC for this project. Parsons will only mobilize to a property after an access agreement has been negotiated and signed by each property owner and a date and time has been scheduled for sampling by the DTSC. The Parsons Field Team will maintain a copy of each access agreement in the field. A Parsons representative, in conjunction with a DTSC representative, will contact each residential occupant prior to the scheduled start of field activities to ensure that each is aware of the project schedule and anticipated activities. If any questions or concerns are raised by the occupant, the DTSC Project Manager will be contacted. At some properties, the owner may not be on site and renters may be present.

3.2 Utility Clearance

Prior to the start of intrusive work, a number of steps will be taken to prepare for the field activities. The initial reconnaissance will include a field check for any utilities or landscape irrigation lines. These can be identified by locating water valves, irrigation sprinklers, and gas and electric meters. Because no intrusive work other than hand augering is expected, a subsurface utility survey will not be conducted. At least 48 hours before intrusive field tasks begin, Underground Services Alert (USA) will be notified of the intent to conduct subsurface investigations.

3.3 Sampling

3.3.1 Soil Screening with XRF

Soil Sample Location Selection and Sample Collection

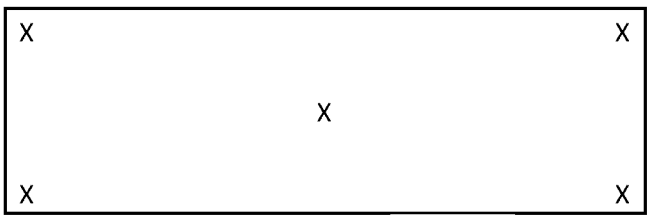
The following steps will be taken to select the soil sampling areas:

1. Sampling locations will target bare exposed soils that have not been recently disturbed and open grassy areas away from structures or thick trees. Sampling locations will target areas, including play and garden areas, in which maximum deposition and exposure potential are likely.
2. To ensure that the sampling locations represent locations of maximum aerial deposition, soil will not be collected in the following areas: within areas that were recently disturbed; within 2 ft of a roadway; within 5 ft of potential property-specific contamination sources (e.g., trash, burning areas, waste storage areas,

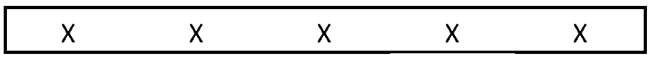
etc.); beneath crushed stone, dirt or gravel driveways, or parking areas; and from public areas.

3. The area for sampling will be selected using the following criteria listed in order of importance: outside the exclusionary criteria in Section 3; bare, exposed soils; open grassy areas; child play areas; and garden areas.

4. Approximately 15 sample locations will be selected at each property; each location will be marked with pin flags. The locations will be as evenly spaced as possible to achieve coverage of the area with preference for bare soils. If a designated play area is on the property, two additional soil samples will be collected from the play area for a total of 17 sample locations. For example, a square or rectangular yard area would be sampled as follows:



A thin, rectangle-like tree lawn would be sampled as follows:



In most cases, the 15 soil sampling locations will be distributed as follows: five locations in the front yard; five locations in the back yard; five locations distributed in drip zones, near downspouts, side yards, and other open bare soils areas; and two additional contingency sample locations if a play area is present.

5. Soil samples will be collected at all locations for the 0- to 3-inch depth interval. In the two highest detected lead concentration locations, samples will be collected from three additional discrete depth intervals at 3- to 6-inch, 6- to 12-inch, and 12- to 18-inch depth. All depth intervals will be screened with the XRF analyzer at all locations for a total of up to 23 XRF soil sample analyses per property.

6. If grass is present at the sample location, the grass and root mat will be carefully cut away and removed. Loose dirt will be shaken into the plastic Ziploc bag for the 0- to 3-inch depth interval sample. The grass will be set aside to be replaced after sampling is complete.

7. Prior to sample collection, an insitu soil moisture reading will be taken near surface. Moist soil samples will be allowed to either air dry, or they will be dried with a portable gas camping stove until a moisture content of less than 20 percent is achieved.

8. Soil from each depth interval will be placed into separate new plastic Ziploc bags. Lumps, rocks, or grass that could interfere with the XRF readings will be removed. The sample will be homogenized in the Ziploc bag for 1 to 2 minutes.

9. After sample homogenization, and in accordance with EPA Method 6200 (EPA, 2007), the sample will be sieved through a Number 60 mesh sieve (250 microns).

10. After sample sieving, the sieved soil will be placed back into a Ziploc bag for XRF data collection.

11. At the two locations where deeper soil samples are collect, a measuring tape will be used to confirm that at least 18 inches of sample was retrieved. Any material extending beyond 18 inches, or slough collected in the hand auger bucket will be returned to the site where it was originally collected.

12. All reusable equipment, such as hand trowels, sieves and bucket augers, will be decontaminated. Gloves will be changed between sampling intervals. All particulate matter and surface films will be removed with water. Reusable sampling equipment will be first washed in a water/Alconox solution and then rinsed with clean water. Decontaminated equipment will be properly covered and stored prior to use at the next sampling location to prevent cross-contamination.

13. The location of each sample will be measured from a reference point at the property and marked on a field sketch. In addition, coordinates of each soil sampling location will be recorded using a global positioning system (GPS) unit. GPS coordinates of each sampling location will also be recorded in the field notes.

These field procedures may be modified based on the soil conditions encountered. If paint chips from onsite structures are visible within the drip line, they will be collected in plastic bags, described accordingly with photographs, and submitted for laboratory analysis. Sampling locations near potential presence of non-aerial depositional sources such as stains, debris, burn pits, or peeling paint will also be carefully documented in notes and by photograph.

XRF Analysis of Soil Samples

All soil samples will be analyzed in the field using XRF methods as described in EPA Method 6200 (EPA, 2007). A copy of EPA Method 6200 is provided in Appendix B.

The use of field portable XRF will be the primary method of estimating lead in affected soils in the field for screening and verification purposes. However, the field portable XRF method has a distinct operating range and is subject to interferences caused by site-specific physical and chemical characteristics of the sample, which must be understood in order to optimize the use of the instrument. These interferences include the following:

- Physical matrix effects, such as variations in particle size and sample homogeneity
- Sample moisture content greater than about 20 percent
- Inconsistent positioning of samples in front of the probe window
- Chemical matrix effects resulting from differences in the concentrations of interfering elements
- Changes in ambient air temperature producing instrument drift.

EPA Method 6200 (EPA, 2007) is a standard analytical method that guides the use of field portable XRF instruments. The method discusses the two modes in which field portable XRF instruments can be operated: in situ and intrusive. The in situ mode involves analysis of an undisturbed soil. Intrusive analysis involves collection and preparation of a soil sample before analysis. In situ analysis is an attractive method in that no sample is collected and prepared, only limited preparation of the surface to be sampled is needed, and results can be obtained

rapidly. In practice, however, in situ results can be highly variable (order of magnitude) and subject to most, if not all, of the interferences noted above. In addition, in situ measurements could damage an expensive instrument and expose the unit to dirt and possible contamination. Therefore, in situ measurements will not be used on this project.

The preparation methods with the XRF analysis through the sample bag have certain disadvantages, including attempting analysis through the thicker plastic sample bag and placing the analyzer window in an optimal position. Sample results are also more difficult to reproduce. However, in the case of the sample bag method, an analysis can be performed quickly, which may be useful for sample screening (e.g., identifying samples with extremely high concentrations where no further analysis would be required).

The XRF device will be calibrated daily and operated by a trained individual who is certified in California to use a field-portable XRF. To confirm that the XRF is within allowable tolerances, the XRF will conform to the National Institute of Standards and Technology (NIST) standards (NIST 180-661 and 180-673) prior to its use in the sampling. The concentrations of the metals and analysis of standards will be determined daily and will be recorded on the daily worksheet.

Prior to soil sample collection, an insitu soil moisture reading will be collected at ground surface (0-3"). The hand-auger or trowel sample will be placed directly into a new, unused plastic Ziploc bag that will be discarded after one use. Soil samples will be prepared for XRF analysis by homogenizing within the plastic bag. Large soil particles will be broken up by hand in order to create a homogenous material suitable for XRF analysis through the bag. Moist soil samples will be allowed to either air dry, or will be dried using a gas camping stove if moisture content is above 20%. After proper moisture content is achieved, the sample will be sieved through a No. 60 screen. After proper homogenization and preparation, the sample identification will be entered onto a XRF worksheet along with the XRF reading results, the testing date and times, the run time (30 seconds minimum), and the (corrected and uncorrected) metals result(s). Standard check results will also be entered on the worksheet. The worksheet will also note if a sample was sent to the off-site analytical laboratory for analysis. A sample worksheet is provided in Attachment B. Copies of the completed worksheets will be provided in the subsequent Soil Sampling Report.

Following the first XRF reading, a minimum of four additional readings will be performed on four additional locations of the sample bag and recorded on the worksheet. The results for metals (antimony, arsenic, cadmium, lead, copper, and zinc) will be entered onto the XRF worksheet along with the testing duration. If a specific analyte is below the detection limit (DL), the DL will be entered onto the worksheet in order to calculate an average for the analyte.

Research on reproducibility of XRF data indicates that longer XRF reading times resulted in better correlation and reproducibility. Therefore, the team will follow the above sampling procedure for the first 10 residences. The reproducibility of the data and a comparison of the averages produced from five data points collected from an individual sample will allow us to determine if this lengthy procedure is warranted. If warranted, we will modify this protocol in consultation with the DTSC.

The XRF correction factors and summary tables of corrected XRF data will be provided in the subsequent report. This information will be used in conjunction with laboratory results to create profiles that will be used to guide the Remediation Contractor through soil removal activities. Laboratory samples will be analyzed for lead, copper, zinc, antinomy, and cadmium by EPA Method 6010B. A Certificate of Registration for the XRF device to be used for the soil sampling will be obtained from the State of California Health and Human Services Agency, Department of Public Health (CDPH), prior to its use in the field. A copy of the Certificate of Registration, all completed registration forms, and CDPH approval letter will be included in the subsequent report. The CDPH will also be notified of the mobilization/demobilization of the XRF within the appropriate time periods set forth by the CDPH, with copies of all notices to be provided in the Report.

3.3.2 Soil Laboratory Sample Collection

Following the XRF analysis described above, 10 percent of the soil samples with the highest lead concentrations from each property will be submitted for fixed laboratory analysis (approximately two samples per property). Soil samples will be transferred from the Ziploc bags used for XRF analysis to new glass jars provided by the laboratory. Each jar will be labeled with the corresponding sample identification (ID), time, date, project name, and client name. All soil samples will be bubble wrapped, placed in Ziploc bags, and stored under ice in a cooler. The soil samples will be submitted to a designated analytical laboratory under a chain-of-custody (COC) record. The laboratory will be certified in the state of California and certified by the Environmental Laboratory Accreditation Program (ELAP). All soil samples will be analyzed for lead, copper, zinc, antinomy, and cadmium using EPA Method 6010B. Soil samples will be analyzed with no more than a 2-week turnaround time. Standard Level 1 electronic data packages will be provided by the laboratory. The laboratory will retain all samples until the data evaluation is complete.

Quality Assurance / Quality Control

Parsons will utilize its quality assurance project plan (Parsons 2015b) which has set forth all required guidelines for all activities, products, and services and is designed to ensure that all activities are accomplished in an approved, prescribed manner by technically trained and competent staff. At least 10 percent of the total daily soil samples will be submitted as field duplicate samples to determine the precision of the sampler and the analytical laboratory. Duplicate samples will be prepared in the same manner as other samples and will be given the sample designation "D" to indicate that it is a duplicate sample. Field duplicate samples will be analyzed lead, copper, zinc, antinomy, and cadmium by EPA 6010B.

Equipment Blanks

Equipment blanks will be prepared when a particular piece of sampling equipment was employed for sample collection and subsequently decontaminated in the field for use in additional sampling. The equipment blank will be taken in the field by collecting a blank water rinse from the equipment (e.g. hand auger bucket) in the appropriate pre-preserved container after execution of the last step of the field decontamination protocol. One equipment blank will be collected per team for each day of testing. Each equipment blank will be analyzed for lead, copper, zinc, antinomy, and cadmium by EPA Method 6010B.

Matrix Spike/Matrix Spike Duplicate Samples

The laboratory will split matrix spike/matrix spike duplicates (MS/MSD) from one sample collected from each sampling day and will analyze the sample for the same parameters as the parent sample. Each sample will be labeled with the sample identification as the original sample and will be designated as MS or MSD samples. MS/MSD samples determine accuracy by the recovery rates of the compounds added by the laboratory (the MS compounds are defined in the analytical methods). The MS/MSD samples also monitor any possible matrix effects specific to samples collected from the Site and the extraction/digestion efficiency. In addition, the analyses of MS and MSD samples check precision by comparing the two spike recoveries.

Data Analysis

Following receipt of the electronic data packages, a Level 1 review will be conducted. This review includes checks on holding times, blank contamination, MS/MSD results and duplicate analysis, and completion of the associated checklist. The results will be compiled into Excel spreadsheets for data presentation and analysis.

3.3.3 LBP Testing

The LBP testing for this sampling effort is proposed as a preliminary screening approach. No published strategies currently exist for field XRF testing at commercial, industrial, school, public buildings, or soil testing. The procedures for the LBP testing of the exterior of the structures in remedial areas will not follow the Department of Housing and Urban Development (HUD) guidelines for LBP testing. The intent is to provide a screening of potential LBP on the exterior of buildings, if paint is in a deteriorating state, and to the extent that it might affect the nearby soil. Therefore, the surveyor will use available information, experience, and judgment, together with XRF technology, to develop a testing strategy and provide information about potential presence of LBP on the exterior of the buildings only if paint is in a deteriorating state. The following criteria will be used to perform the LBP testing:

- ***Color.*** Lead is added to paints for pigmentation and corrosion resistance. Parsons assumes that paints of similar color contain similar amounts of lead and, therefore, will test each color observed.
- ***Substrate.*** Lead is used as a primer for various substrates. However, similar to topcoats, the undercoat primer and other paint layers could be different. It is assumed that, on each substrate type in the building (e.g., metal, wood, wallboard, and stucco), primer and undercoat paint are consistently applied and contain similar quantities of lead, if any. Thus, each substrate observed will be tested.
- ***Building Components.*** Building components (e.g., walls, floor, and ceiling) could have been painted with different colors of paint throughout the history of the building. It is assumed that the different components had different primers and undercoats applied even though the topcoat colors appeared similar. It is also assumed that similar primer and paint had been applied underneath the top layer on similar building components. Thus, each building component observed will be tested.

- ***Functional Areas***. A functional area consists of a group of areas put to similar use where similar topcoats of paint are observed (e.g., exterior walls). Because the primer and paint in the same functional area probably contain similar amounts of lead, each functional area will be tested rather than every individual area within.

Up to six XRF readings are proposed for exterior structures in case peeling and deteriorating paint is observed. Only if destructive sampling is not required, or the access agreement allows for collection, paint chips from exterior of structures within each property will be collected for laboratory analysis by EPA Method 6010B. XRF data from each residence will be recorded on the field data sheet presented in Appendix C.

XRF Data Evaluation Criteria

When an XRF analyzer is used to test painted surfaces, the HUD guidelines and Los Angeles County (LAC) Health and Safety Codes specify action levels (ALs) of 1.0 and 0.7 milligrams per square centimeter (mg/cm^2), respectively. Because the properties are located in LAC, 0.7 mg/cm^2 will be used to evaluate the presence/absence of LBP on various building components.

The performance characteristic sheet (PCS), as specified by HUD (Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing, 2012 Edition), provides an inconclusive range for each type of XRF analyzer and is only relevant at the AL of 1.0 mg/cm^2. The same inconclusive range is not available or applicable for the more stringent LAC AL of 0.7 mg/cm^2. Because of the limitations of field portable XRF analyzers, an "inconclusive" range of 0.6 to 0.8 mg/cm^2 is arbitrarily established and used for this screening.

Because the number of locations tested is limited by practical considerations, certain painted surfaces judged to pose a minimal potential hazard during remediation or impact to the nearby soil will be excluded from the survey. These surfaces include miscellaneous artwork, graffiti, trash, debris, some areas smaller than 10 square feet (ft^2), movable fixtures (e.g., chairs, tables, lights, and cabinets), and building components that can be removed with little or no disturbance to the LBP.

Terminology

The 1997 HUD guidelines originally defined terms "intact," "fair," and "poor" referring to the conditions of LBP observed at the time of the survey (HUD, 1997). In the 2012 revised version of the HUD guidelines, additional terms describing LBP conditions were used including "good condition," "*de minimis* (minimal) amount," and "deteriorated condition" (HUD, 2012). Because the DPH has not adopted HUD 2012 definitions and for clarification purposes in this report, the following definitions are qualitatively applied within the framework of Parsons' judgment and the modified version of definitions in the 1997 and 2012 HUD guidelines:

- Intact: Paint generally in good condition
- Fair: Paint generally intact with minor, normal wear and tear; or *de minimis* amount of damage at:
    - Less than 20 ft^2 on exterior surfaces,

- Less than 10 percent of the total surface area on the exterior component type of a small surface area (i.e., window sills, baseboards, trims, etc.).

- Poor: Paint not intact, severely worn, damaged, chalking, or deteriorated; or damaged beyond the *de minimis* amount.

For discussion purposes, the term "LBP" will be used for or defined as any paint reported to contain lead concentrations greater than or equal to 0.7 mg/cm^2 as determined by the field XRF analyzer.

Typically, three classifications are used for results: positive, inconclusive, and negative. A positive classification is defined as LBP at or above 0.7 mg/cm^2. Negative and inconclusive classifications, which are based on the PCS as published by each manufacturer, are substrate-dependent. When no inconclusive reading was recorded, a negative classification is defined as any paint reported to contain less than 0.7 mg/cm^2.

3.3.4 Sample ID Designation

Samples will be identified first by a unique property number and a unique sample identification number. Soil samples will also include the bottom depth of the sampling interval. The following is an example of the sampling nomenclature:

XRF and Laboratory Soil Samples

(Property Number – Sample Number - Bottom Depth of Sample Interval)

PIA0001-01-03 (for 0 to 3 inches)

PIA0001-01-06 (for 3 to 6 inches)

PIA0001-01-12 (for 6 to 12 inches)

PIA0001-01-18 (for 12 to 18 inches)

XRF and Laboratory Paint Samples

(Property Number – Sample Number)

PIA0001-01-LBP

Duplicate samples will be collected for samples submitted to the laboratory. All duplicate samples will be identified with a "D", for example, PIA0001-01-3D.

Other quality assurance samples will have the following IDs:

Tripblanks – (*TP-Property Number-Date*) TP-PIA0001-111715

Equipment Blanks – (*EB-Property Number-Date*) EB-PIA0001-111715

Field Blanks – (*FB-Property Number-Date*) FB-PIA0001-111715

3.3.5 Sampling Equipment

The following or similar appropriate equipment will be used for soil sampling:

- A Niton XU 700 Series XRF analyzer

- A 2-inch-diameter bucket auger
- Stainless steel trowel
- Chisel for scraping paint chips into a plastic bag
- Small and large plastic Ziploc Bags
- Paper towels
- Disposal gloves
- Samples glass jars and labels
- Coolers and ice

3.3.6 Documentation

Field Logbooks

Field logbooks will document where, when, how, and from whom vital project information was obtained. Logbook entries will be complete and accurate enough to permit reconstruction of field activities. Logbooks will be bound with consecutively numbered pages. Each page will be dated and the time of entry noted in military time. All entries will be legible, written in black ink, and signed by the individual making the entries. Language will be factual, objective, and free of personal opinions or other terminology that might be inappropriate. If an error is made, corrections will be made by crossing a line through the error and entering the correct information. Corrections will be dated and initialed. No entries will be erased or rendered unreadable.

At a minimum, entries in the field logbook will include the following information for each sample date:

- Project name and address
- Recorder's name
- Team members and their responsibilities
- Time of arrival/entry onsite and time of departure
- Other personnel onsite
- Summary of any onsite meetings
- Deviations from sampling plans and site safety plans
- Changes in personnel and responsibilities as well as reasons for the changes
- Levels of safety protection
- Calibration readings, equipment model, and serial number for any equipment used

At a minimum, the following information will be recorded during the collection of each sample:

- Sample identification number
- Sample location and description
- Sketch showing sample location and measured distances
- Sampler's name(s)
- Date and time of sample collection
- Designation of sample as composite or grab

- Type of sample (i.e., matrix)
- Type of preservation
- Type of sampling equipment used
- Field observations and details important to analysis or integrity of samples (e.g., heavy rains, odors, and colors)
- COC form numbers and seal numbers
- Transport arrangements (e.g., courier delivery or lab pickup)
- Recipient laboratory

Field XRF Sheets

All XRF data will be recorded on the field data sheets presented in Appendix C.

Chain-of-Custody Records

COC records are used to document sample collection and shipment to the laboratory for analysis. All sample shipments for analysis will be accompanied by a COC record. Form(s) will be completed and sent with the samples for each laboratory and each shipment. If multiple coolers are sent to a single laboratory on a single day, separate COC form(s) will be completed and sent with the samples for each cooler. The COC record will identify the contents of each shipment and will maintain the custodial integrity of the samples. Generally, a sample is considered to be in someone's custody if it is either in someone's physical possession, in someone's view, locked up, or kept in a secured area that is restricted to authorized personnel. Until the samples are received by the laboratory, they will be the responsibility of the sample collector.

Photographs

Photographs will be taken at selected sample locations and at other areas of interest onsite. They will serve to verify information entered in the field logbook. When a photograph is taken, the following information will be written in the logbook or will be recorded in a separate field photography log:

- Time, date, location, and (if appropriate) weather conditions
- Description of the subject photographed
- Name of person taking the photograph

Sketches

Sketches will be produced in the field detailing the exact location of each soil and LBP sampling locations. A sketch will be produced for each property and it will contain at a minimum the following information:

- An approximate layout of the property with dimensions, and the relation to the street
- Sampling locations with measurements from a reference point
- A unique property number, address, date, and initials of the employee that created the sketch.

4 REPORTING AND DELIVERABLES

Sampling reports will be provided for each property. Sampling reports will include, but are not limited to:

- A description of the property
- A map showing the sampling locations
- Coordinates of the sampling locations
- Sampling results in tabular form and electronic format (MS Excel)
- Screening of the results against criteria established in the Workplan to determine if further action is required at the property
- Photographs of the sampling locations
- Laboratory analysis reports
- An evaluation of the quality of the data
- An explanation of any deviation from the Workplan

Sampling reports will be submitted within 30 days from the sampling event and will be signed and stamped by a professional engineer or geologist. Sampling of 1,000 properties will be completed no later than May 30, 2016, so that all activities listed in this workplan are completed no later than December 30, 2016.

5 REFERENCES

EPA, 2007. *Method 6200, Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment*, Department of Toxic Substances Control. February 2007.

U.S. Department of Housing and Urban Development (HUD), 1997. *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing.* Revised 1997.

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Parsons, 2015a. Exide Technologies – Off-site Remediation and Restoration, Project Safety, Health, and Environmental Plan. Novmeber 2015.

Parsons, 2015b. Quality Assurance Project Plan. Exide Facility (Vernon, California). November 2015.

U.S. Department of Housing and Urban Development (HUD), 2012. *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing, Second Edition.* July 2012. http://portal.hud.gov/hudportal/HUD?src=/program_offices/healthy_homes/lbp/hudguidelines

Figures

FIGURE 1

Northern and Southern Assessment Areas Location Plan

Off-Site Soil Sampling Work Plan Addendum

DTSC Exide Site

PARSONS

Pasadena, CA

K:\Depts\Dept42b\DTSC\Orphan Sites MSA\Exide\03 Offsite sampling Workplan – 1000 homes\Figure 1 – Northern & Southern Assessment Areas.dwg

APPENDIX A
QAPP and DQOs

QUALITY ASSURANCE PROJECT PLAN (QAPP) FOR SAMPLING AND ANALYSIS OF PROPERTIES IN THE VICINITY OF THE EXIDE FACILITY (VERNON, CALIFORNIA)

Prepared for

The Department of Toxic Substances Control
8800 Cal Center Drive
Sacramento, CA 95826

Prepared by

PARSONS

100 WEST WALNUT STREET
PASADENA, CALIFORNIA 91124

November 18, 2015

QUALITY ASSURANCE PROJECT PLAN (QAPP) FOR SAMPLING AND ANALYSIS OF PROPERTIES IN THE VICINITY OF THE EXIDE FACILITY (VERNON, CALIFORNIA)

Prepared For:

Department of Toxic Substances Control

Prepared By:

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Reviewed by:

11/18/15

Cindy Zicker, Project Chemist

Reviewed by:

11/18/15

Jim Goepel, Project Technical Director

Reviewed by:

11/18/15

Shala Craig, PE #C-69804, Project Manager

DTSC EXIDE SITE
QUALITY ASSURANCE PROJECT PLAN

REVISION HISTORY

Revision No.	Date	Revised By	Reason for Revision	Sections Revised
0	11/18/15		Original Document	All

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LIST OF ATTACHMENTS

ACRONYMS AND ABBREVIATIONS

AL	Action Level
ASTM	American Society for Testing and Materials
Cal-EPA	California Environmental Protection Agency
CCR	California Code of Regulations
CFR	Code of Federal Regulations
°C	degrees Celsius
COC	chain-of-custody
COPC	chemical of potential concern
CDPH	State of California Health and Human Services Agency, Department of Public Health
DF	dilution factor
DL	detection limit
DQA	data quality assessment
DQO	data quality objective
DTSC	Department of Toxic Substances Control
ELAP	Environmental Laboratory Accreditation Program
FM	field manager
ft	feet
ft^2	square feet
GPS	global positioning system
HASP	Health and Safety Plan
HUD	Department of Housing and Urban Development
IMWP	Interim Measures Workplan
J	estimated concentration
LAC	Los Angeles County
LCS	laboratory control sample
LBP	lead-based paint
MDL	method detection limit
mg/kg	milligrams per kilogram
mg/cm^2	milligrams per square centimeter
MS/MSD	matrix spike/matrix spike duplicate
NC/CAR	non-conformance/corrective action report
NIST	National Institute of Standards and Technology
OEHHA	Office of Environmental Health Hazard Assessment
OSHA	Occupational Safety and Health Administration
PAH	polynuclear aromatic hydrocarbon
PARCC	precision, accuracy, representativeness, completeness, and comparability
PC	percent complete
PCB	polychlorinated biphenyl
PE	performance evaluation
PM	project manager
POC	point of contact
PQL	practical quantitation limit
QAP	quality assurance plan
QAPM	quality assurance program manual

QAPP quality assurance project plan
QA/QC Quality Assurance/Quality Control
RFD relative percent difference
SD sample duplicate
SI Site Investigation
SOP standard operating procedure
SOW scope of work
SQL sample quantitation limit
SCAQMD South Coast Air Quality Management District
SVOC semi-volatile organic compound
TPH-d total petroleum hydrocarbons (diesel-range organics)
TPH-g total petroleum hydrocarbons as gasoline-range organics)
TPH-o total petroleum hydrocarbons as gasoline (oil-range organics)
U non-detect
USA Underground Services Alert
USEPA United States Environmental Protection Agency
VOC volatile organic compound
Workplan Site Characterization Workplan
XRF X-ray fluorescence

SECTION 1.0
INTRODUCTION

This Quality Assurance Project Plan (QAPP) has been prepared to support site assessment and remediation activities being conducted for the California Environmental Protection Agency (Cal-EPA) Department of Toxic Substances Control (DTSC) for residential and sensitive-use properties located in the vicinity of the Exide Metals facility (site) in Vernon, California. The purpose of this QAPP is to present the organization, objectives, functional activities, and specific quality assurance (QA) and quality control (QC) activities in support of anticipated sampling activities.

This QAPP incorporates the following references in establishing the project criteria:

- United States Environmental Protection Agency (USEPA), *Guidance for the Data Quality Objectives Process* (USEPA, 1994b);
- USEPA, *Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, SW-846, Third Edition, Update III* (USEPA, 1996);
- American National Standards Institute/American Society of Quality Control (ANSI/ASQC E-4-1994), *Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs*, July 1995; and
- USEPA, *Risk Assessment Guidance for Superfund, Volume 1: Human Health Evaluation Manual* [Parts A, B, and C] (USEPA, 1989, 1991a, and 1991b).

The procedures described herein will be performed in accordance with the guidance, regulations, and documents presented in the project statement of work.

1.1 PROJECT DESCRIPTION

Parsons has been tasked by the DTSC with the investigation and cleanup of residential and sensitive-use properties located near the former Exide Technologies (Exide) battery recycling facility in Vernon, California. Lead emissions from the former Exide facility are suspected of affecting surface and near-surface soils in surrounding areas as a result of aerial deposition.

The initial phase of assessment work will evaluate soil lead concentrations at up to 1,000 residential and sensitive-use properties to ascertain the need for soil removal. The goal of this investigation is to identify those residential properties that contain lead soil concentrations equal to or greater than 1,000 mg/kg. Properties with these lead concentrations in soil are considered having the greatest lead exposure potential and will be prioritized for cleanup. Field analyses will be performed using an X-ray fluorescence (XRF) analyzer to allow for rapid evaluation of multiple properties; confirmation sampling of a selected subset of samples will be performed by an off-site fixed laboratory.

This QAPP is designed to support both site characterization and remedial action activities. The scope of work for site characterization sampling will primarily focus on the collection of soil samples for metals (primarily lead) analysis, although other metals may also be targeted during sampling activities. In addition, limited XRF field screening

of painted surfaces for the presence of lead-based paint (LBP) will also be performed. The scope of work for remedial action activities is to collect confirmation soil samples and waste profiling samples.

1.2 PURPOSE AND SCOPE OF QAPP

This QAPP sets forth quality guidelines for all activities, products, and services and is designed to ensure that all activities are accomplished in an approved, prescribed manner by technically trained and competent staff. This document establishes the QA requirements and assigns responsibility to project personnel and subcontractors for ensuring that project objectives will be achieved. This QAPP consists of the QA program requirements that are responsive to all guidance documents referenced in Section 1. Quality requirements specified in this document are tailored to the needs of this assessment project.

1.3 PROJECT OBJECTIVE

The objective of the assessment work is to characterize the presence of lead in soil at multiple off-site residential and sensitive-use properties to determine if aerially deposited lead may be present at concentrations of potential concern from a human health perspective. The objective of the remediation work is to remove lead-impacted soils at those properties that represent a potential threat to human health, ensure lawful disposal of the removed soils, and perform site restoration.

1.4 PROJECT ORGANIZATION AND RESPONSIBILITIES

This project will be executed with Parsons personnel and various subcontractors. Subcontractors will include the analytical laboratory and the remediation contractor. The responsibilities of the positions relevant to project QA/QC are summarized below.

DTSC Contract Management Representative

Mr. Raymond Leclerc, PE, of the DTSC is responsible for overall coordination and organization of the Exide project, including this investigation work. He can be reached at (916) 255-3528. Ray may delegate authority to DTSC field representative for field-related decisions.

DTSC Project Manger

Mr. Peter Ruttan, P.G. will represent the DTSC. He will review and approve the Workplan and will coordinate all environmental activities with Parsons. He can be reached at (916) 255-3630

Parsons Project Manager

Ms. Shala Craig, PE is Parsons' Project Manager for providing environmental services to the Design Team. In this capacity, she will be the primary liaison between the DTSC and Parsons. She can be reached at (310) 612-3393. The Parsons PM reports directly to the DTSC PM and exercises control over all project activities including field investigation, remedial action, and report writing activities. The Parsons PM is ultimately responsible for planning and staffing to meet project requirements, assuring adequate planning and execution of the health and safety plan, implementing the QAPP, by overseeing analytical

data quality, data management and project requirements. The Parsons PM is also responsible for budget, schedule, and quality of technical memoranda, data packages, and reports.

Parsons Technical Manager

Mr. Jim Goepel is Parsons' Technical Director and will be responsible for all field work coordination. He can be reached at (626) 440-6013. The Technical Manager reports to the Project Manager and provides support in terms of ensuring overall technically adequacy of approaches, maintaining oversight of sampling and analysis activities, and performing technical review of deliverables, and coordination of other technical issues that may arise on the project.

Parsons Field Manager (FM)

The FM exercises project oversight of the field investigation/remedial action activities and reports to the project manager. The FM oversees the day-to-day progress of the investigation/remedial action, including manpower, scheduling, and compliance with the QAPP. The FM is also responsible to the PM for the conduct of site investigation/remedial action activities and the coordination and scheduling of subcontract support. Responsibilities of the FM include the following:

- Supervising the field team, including field geologists, technicians, and subcontractors;
- Correcting non-conformance issues identified in field methods;
- Implementing field health and safety protocols, and interacting in field procedure training for all newly assigned field personnel; and
- Ensuring compliance with the QAPP in handling and recording field samples.

Parsons QA Officer

The QA Officer reports to the Parsons PM and coordinates directly with the FM. The Project QA Officer is responsible for ensuring that sufficient QA procedures are developed for the project, that adequate quality controls are imposed to achieve the required level of QC and that the controls are implemented properly. Responsibilities of the QA Officer include the following:

- Ensuring that project-required QA/QC procedures are clearly specified for field and laboratory activities;
- Working directly with the PM, field personnel, and the laboratory's PM to ensure that chemical data collection and analytical procedures are adequate for the project-specified level of data quality;
- Ensuring that system and performance audits are routinely performed by the subcontract laboratory;
- Acting as the PM's point of contact with the subcontract laboratory; and
- Ensuring adequate project preparation, quality review, and submittal of the data quality assessment (DQA) report.

Project Chemist

The Project Chemist reports to the PM and is responsible for implementation of the QAPP. Responsibilities of the Project Chemist include the following:

- Ensuring that project-required QA/QC procedures are clearly specified for field and laboratory activities;
- Working directly with the PM, field personnel, and the Laboratory's PM to ensure that chemical data collection and analysis procedures are adequate for the project-specified level of data quality;
- Ensuring that timely audits of the subcontract laboratory are performed;
- Ensuring adequate project preparation, quality review, and submittal of the DQA report.

Laboratory QA Officer

The Laboratory QA Officer is responsible for ensuring that sufficient QA procedures are applied to laboratory analyses. The Laboratory QA Officer is also responsible for ensuring that adequate laboratory controls are utilized for a high level of data quality, and that data program requirements and data quality objectives (DQOs) are met. Responsibilities of the Laboratory QA Officer include the following:

- Initiating nonconformance reports and/or corrective actions as necessary;
- Verifying completion of corrective actions for major non-conformances issues cited in audits;
- Reviewing all statistical data to verify that the analytical laboratories are meeting stated QC goals; and
- Coordinating with the Project Chemist and Laboratory PM.

Laboratory Project Manager

The Laboratory PM is responsible for implementation of the QAPP (for analytical control) and any laboratory subcontract. The Laboratory PM ensures that project-required QA/QC procedures for laboratory activities are adhered to for the project-specified level of data quality. The Laboratory PM acts as the primary point of contact between the subcontract laboratory and Parsons.

SECTION 2.0
DATA QUALITY OBJECTIVES

The objective of collecting and analyzing environmental samples for this project is to ascertain the distribution of chemicals of potential concern (COPCs), primarily lead, in surface and near-surface soils at various residential and sensitive-use properties near the Site. At those properties where lead concentrations exceed established thresholds, soil removal and restoration activities will be performed and environmental sampling will be performed to confirm the effectiveness of the cleanup. This QAPP has been developed for use in conjunction with sampling activities to be undertaken at the site, and describes the QA/QC procedures and protocols that will be used during sample analysis. The QAPP will serve as a controlling mechanism during the investigation/remedial action to ensure that a sufficient quantity of data is collected and that all data collected are valid, reliable, and defensible.

An effective QA program addresses DQOs for both field sampling and laboratory methods. The field QA efforts will focus on ensuring that samples are representative of the conditions in the various environmental media at the time of sampling and that the field analytical approach is properly implemented. Both field-based analytical and off-site fixed-based subcontract laboratory QA efforts will be aimed primarily at ensuring that analytical procedures provide sufficient accuracy and precision to reliably quantify contaminant levels in environmental samples. The subcontract laboratory will also ensure that analyzed portions are representative of each sample.

Per USEPA (2000), the DQO process is a seven-step systematic planning process used to develop sampling designs for data collection activities that support decision making. The systematic planning process is applied during the development of a sampling approach using qualitative or quantitative statements to clarify study objectives, define a sampling approach for collecting and analyzing data (e.g., location and number of samples to collect, field sampling methods, analytical methods, etc.), identify critical decision points, determine decision criteria and rules, and specify tolerable levels of potential decision errors. The seven steps of the DQO process are:

1. State the Problem
2. Identify the Decision
3. Identify the Decision Inputs
4. Define the Boundaries of the Study
5. Develop Decision Rules
6. Specify Tolerance Limits on Decision Errors
7. Optimize the Design for Obtaining Data

The DQO process was applied during the development of the soil sampling approach and is summarized in Table 1. The primary DQO decision question for the soil investigation is to determine if soil concentrations at individual properties exceed the site-specific soil screening level for lead of 80 mg/kg, which is protective of incidental ingestion, dermal contact, and inhalation of particulates (see Step 2 in Table 1).

2.1 ANALYTICAL DATA QUALITY LEVELS

The analytical levels for this project's DQOs will conform to the two USEPA-defined categories of data. These data categories are defined below:

Screening Data - Screening data are generated by more rapid, generally less precise methods of analysis with less rigorous sample preparation. Sample preparation steps may be restricted to simple procedures such as removing non-soil particles (e.g., roots) within the soil matrix. Screening data generally provide less-certain quantification of contaminant concentrations.

Definitive Data - Definitive data are generated using rigorous analytical methods, such as approved USEPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce tangible raw data (e.g., chromatograms, spectra) in the form of hard-copy printouts or computer-generated electronic files. Data may be generated at the site or at an off-site location, as long as the QA/QC requirements are satisfied. For the data to be definitive, either analytical or total measurement error must be determined. Results of fixed-based laboratory analyses of samples collected at the site under this QAPP will be considered definitive data.

Screening data and definitive data quality levels will be used as indicated below:

- Screening analyses will be used for screening air in worker breathing zones for health and safety purposes.
- Screening XRF analyses will be used to rapidly characterize concentrations of lead (and other metals as necessary) in soil at the large number of properties that will be evaluated during anticipated sampling efforts.
- Definitive analyses from an off-site fixed laboratory will be used to confirm the XRF results and provide data to support a performance evaluation study with regards to the accuracy and representativeness of the XRF results.
- Definitive analyses from an off-site fixed laboratory will be used on an as-needed basis to support waste characterization requirements associated with off-site disposal of lead-impacted soils from the remediation phase of the work.

2.2 DATA QUALITY ASSESSMENT CRITERIA

DQA criteria will be used to evaluate the quality of the field sampling efforts, field screening results, and fixed-base laboratory results for compliance with project DQOs. The DQA criteria are expressed in terms of analytical precision, accuracy, representativeness, completeness, and comparability (PARCC). Procedures used to assess data accuracy and precision are in accordance with USEPA's (1996) *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846.*

2.2.1 Precision

Precision is the measure of variability among individual sample measurements under prescribed conditions. The relative percent difference (RPD) between primary and field duplicate samples, laboratory sample duplicate (SD) pairs, and matrix spike/matrix spike

duplicate (MS/MSD) sample results demonstrate the precision of the sampling and/or the analysis within the batch of samples. When the laboratory control sample (LCS) results meet the accuracy criteria (USEPA, 1996), results are also believed to be precise, and represent the historical precision among the sample batches of the laboratory, independent from the sample matrix. This is based on the LCS being within control limits in comparison to LCS results from previous analytical batches of the same methods and matrices. Precision will be expressed in terms of RPD between the values resulting from primary and duplicate sample analyses. RPD is calculated as follows:

$$RPD = \frac{|x_1 - x_2|}{X} \times 100$$

where:

x_1 = analyte concentration in the primary sample,

x_2 = analyte concentration in the duplicate sample, and

X = average analyte concentration of the primary and the duplicate sample = $(x_1 + x_2)/2$

Acceptable levels of precision will vary according to the sample matrix, the specific analytical method, and the analytical concentration relative to the method detection limit (MDL). For field duplicate samples, the target RPDs are $\leq$ 70 percent for soil samples. If the concentration of either duplicate is less than five times the practical quantitation limit (PQL), a control limit of $\pm$ 2xPQL will be compared against the range of the duplicate pair. The laboratory shall have procedures in place for establishing and updating precision control limits. An RPD within the control limit indicates satisfactory precision in a measurement system.

2.2.2 Accuracy

Accuracy is a measure of the closeness of a reported concentration to the true value. Accuracy is expressed as a bias (high or low) and is determined by calculating percent recovery (%R) from MS/MSDs, LCSs, and surrogate spikes. MS/MSD and surrogate spike recoveries indicate accuracy relevant to a unique sample matrix. LCS recoveries indicate accuracy relevant to an analytical batch lot, and are strictly a measure of accuracy conditions in preparation and analysis independent of samples and matrices. The %R of an analyte, and the resulting degree of accuracy expected for the analysis of spiked samples for QC, are dependent upon the sample matrix, method of analysis, and the compound or element being measured. The concentration of the analyte relative to the detection limit of the method is also a major factor in determining the accuracy of the measurement.

Accuracy expressed as %R is calculated as follows:

$$\%R = \frac{A - B}{C} \times 100$$

where:

A = measured concentration in spiked sample,

B = measured sample concentration (without spike), and

C = concentration of spike added.

The laboratory shall have procedures in place for establishing and updating accuracy control limits. Typical control limits for accuracy are based on the historical mean plus or minus three standard deviations.

2.2.3 Completeness

Completeness is defined as the percentage of laboratory measurements judged to be valid on a method-by-method basis. Valid data are defined as all data and/or qualified data considered to meet the DQOs for this project. Data completeness is expressed as percent complete (PC) and should be ≥ 90 percent. The goal for meeting analytical holding times is 100 percent. At the end of each sampling event, the completeness of the data will be assessed. If any data omissions are apparent, new samples will be collected and reanalyzed for the parameter in question, if feasible. In addition, appropriate corrective action will be implemented to ensure that objectives are met in the future. Laboratory results will be monitored as they become available to assess laboratory performance and its effect on data completeness requirements. When appropriate, additional samples will be collected to ensure that laboratory performance meets PC requirements.

PC is calculated as follows:

$$PC = \frac{N_A}{N_I} X100$$

where:

N_A = Actual number of valid analytical results obtained, and

N_I = Theoretical number of results obtainable under ideal conditions.

2.2.4 Comparability

Comparability expresses the confidence with which data from one sample, sampling round, site, laboratory, or project can be compared to those from another. Comparability during sampling is dependent upon sampling program design and time periods. Comparability during analysis is dependent upon analytical methods, detection limits, laboratories, units of measure, and sample preparation procedures.

Comparability is determined on a qualitative rather than quantitative basis. For this project, comparability of all data collected will be ensured by adherence to standard sample collection procedures, standard fixed laboratory analytical methods, standard field measurement procedures, and standard reporting methods, including consistent units. For example, laboratory lead analyses will be performed on the same exact samples that were tested in the field using instant reading methods, such as X-ray fluorescence; or, concentrations will be reported in a manner consistent with general industry practice

In addition, to support the comparability of fixed-base laboratory analytical results with those obtained from previous or future testing, all samples will be analyzed by

USEPA-approved methods, where available. The USEPA-recommended maximum permissible sample holding times (Table 2) for organic parameters will not be exceeded. Whenever EPA methods are not appropriate or available, recognized methods published by American Standard for Testing and Materials (ASTM) or other recognized organizations with appropriate expertise will be used.

All analytical standards will be traceable to standard reference materials. Initial instrument calibrations shall be first order linear, and shall be checked at the frequency specified for the methods.

2.2.5 Representativeness

Representativeness expresses the extent to which collected data define site chemical impact. Where appropriate, sample results will be statistically characterized to determine the degree to which the data accurately and precisely represent a characteristic of a population, parameter variation at a sampling point, a process, or an environmental condition. Sample collection, handling, and analytical procedures are designed to obtain the most representative sample possible. Representative samples will be achieved by the following:

- Collection of samples from locations that are most likely to be representative of site conditions (based on site scoping, previous results, statistically random sample, etc.);
- Use of appropriate sampling procedures, including proper equipment and equipment decontamination;
- Use of appropriate analytical methods for the required parameters and adequate practical quantitation limits (PQLs); and
- Analysis of samples within the required holding times.

Sample representativeness is also affected by the portion of each sample chosen for analysis. The laboratory will adequately homogenize all samples prior to taking aliquots for analysis to ensure that the reported results are representative of the sample received. Because many homogenization techniques may cause loss of contaminants through volatilization, homogenization will not be performed for any volatile organic compound (VOC) method analyses.

2.2.6 Sensitivity

The concentration of any one target compound that can be detected and/or quantified is a measure of sensitivity for that compound. Sensitivity is instrument-, compound-, method-, and matrix-specific. The definitions of terms relating to sensitivity and DQOs are presented in Section 6.2.

2.3 LABORATORY QA OBJECTIVES

All laboratory analyses will be performed by a State of California Environmental Laboratory Accreditation Program (ELAP); the selected laboratory will provide a copy of their laboratory quality assurance plan (QAP). The QAP shall define internal laboratory procedures for QA/QC and shall include descriptions of the following:

- QA policies and objectives;
- Organization and personnel;
- Document control;
- Analytical methodology standard operating procedures (SOPs);
- Data generation;
- Sample custody, preservation and tracking;
- Data recording, reduction, review, reporting, and validation for both hard copy and electronic formats;
- Security;
- Documentation of client-specific requirements;
- QA audits;
- QC; and
- Non-conformance/corrective action report (NC/CAR) procedures.

The laboratory QAP must be approved by the Parsons PM prior to the initiation of analyses on this project.

2.3.1 Laboratory Standard Operating Procedures

The laboratory must maintain SOPs for all analytical methods and laboratory operations. The format for SOPs must conform to the following references:

- USEPA (1996) Test Methods for Evaluating Solid Waste, Physical and Chemical Methods, SW846, 3rd Edition, Update IIB, Section One;
- USEPA (1995) "Good Automated Laboratory Practices," in Principles and Guidance to Regulations for Ensuring Data Integrity in Automated Laboratory Operations; and
- USEPA (1992) Quality Assurance Technical Information Bulletin, Creating SOP Documents.

All SOPs must have a unique identification number that is traceable to previous revisions of the same document.

2.3.2 Demonstration of Capability, Analyst Training

The laboratory QA department personnel shall maintain records documenting the ability of each analyst to perform applicable method protocols. Documentation will include an MDL study with other annual and quarterly checks for each method and analyst. In

addition, internal, blind performance evaluation (PE) samples for each method and matrix demonstrating overall laboratory performance must be submitted semi-annually.

2.3.3 Laboratory Internal Audits

At a minimum, the laboratory QA department personnel shall perform an annual internal (systems) audit. The internal audit will document compliance with all QAP methods, policies, and procedures. Corrective action must be implemented where required.

SECTION 3.0
FIELD DATA REDUCTION, VALIDATION, AND REPORTING

The following sections describe calibration of field analytical instruments and field data reporting, validation, reduction, and review.

3.1 FIELD RECORD KEEPING

Bound field logbooks will be maintained by the field supervisor and other team members to provide a daily record of significant events, observations, and measurements during the field investigation/remedial action. All entries will be signed and dated. All information pertinent to the field survey and/or sampling will be recorded in the logbooks. The logbooks will be bound, with sequentially numbered pages. Waterproof ink will be used in making all entries. Entries in the logbook will include, at a minimum, the items listed below:

General information:

- Names and titles of author and assistants;
- Date and time of entry;
- Physical/environmental conditions during field activity; and
- Purpose of sampling activity.

In order to provide complete documentation of the sampling event, detailed records will be maintained by the field sampling crew. At a minimum, these records will include the following information:

- Sample location (e.g., street address);
- Sample identification;
- Sample location map or detailed sketch (including GPS coordinates);
- Date and time of sampling;
- Sampling method;
- Field observations of sample appearance and sample odor;
- Weather conditions;
- Sampler's identification;
- XRF readings; and
- Any other relevant information (e.g., moisture content).

3.2 CALIBRATION PROCEDURES AND FREQUENCY FOR FIELD TEST EQUIPMENT

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated according to manufacturer's specifications with sufficient frequency to ensure accuracy and reproducibility of results. At a minimum, monitoring equipment used in the field, including the XRF and dust meters, will be calibrated daily against a known standard. If the results show that the concentration is within 5 percent of the known standard, the equipment will be considered calibrated.

3.3 REVIEW OF FIELD RECORDS

Field record review is an ongoing process. Field team leaders will be responsible for ensuring that proper documentation is recorded during each site's sampling activities. Field records include logbooks, log forms, and any documentation, whether electronic or hardcopy, that is used to record data, observations, assumptions, or other information in the field. The sections below describe the items used for evaluation.

3.3.1 Completeness of Field Records

The check of field record completeness will ensure that all requirements for field activities in the work plan have been fulfilled, complete records exist for each field activity, and the procedures specified in the work plan (or approved as field change requests) are implemented. Field documentation will ensure sample integrity and provide sufficient technical information to recreate each field event. The results of the completeness check will be documented, and environmental data affected by incomplete records will be identified in the technical report.

3.3.2 Identification of Valid Samples

The identification of valid samples involves interpretation and evaluation of the field records to detect problems affecting the representativeness of environmental samples. For example, field records can indicate if unanticipated environmental conditions were encountered during field activities. Records should note sample properties such as clarity, color, and odor. Photographs may show the presence or absence of obvious sources of potential contamination (during sampling). Judgments of sample validity will be documented in the technical report, and environmental data associated with any poor or incorrect field work will be identified.

3.3.3 Identification of Anomalous Field Test Data

Anomalous field data will be identified and explained to the extent possible. Anomalous data will be assessed for usability and explained in the technical report.

3.3.4 Accuracy and Precision of Field Data and Measurements

The assessment of the quality of field measurements will be based on instrument calibration records and a review of any field corrective actions. The accuracy and precision of field measurements will be discussed.

3.4 FIELD DATA VALIDATION

Screening data will constitute all analytical method results from analyses performed in a field laboratory environment including XRF analyses. The Project Chemist will determine if DQOs for field data have been met, and also will calculate the percent complete (PC) for field data results.

At a minimum, the review of screening data will focus on the following topics:

- Holding times;
- Method blanks;

- Field instrumentation calibration and detection limits; and
- Completeness of data.

Field data will be validated using the procedures described below:

- Routine checks (e.g., looking for errors in identification codes) will be made during the processing of data.
- Internal consistency of a data set will be evaluated. This step will involve plotting the data and testing for outliers.
- Checks for consistency of the data set over time will be performed. This can be accomplished by comparing data sets against gross upper limits obtained from historical data sets, or by testing for historical consistency. Anomalous data will be identified.
- Checks may be made for consistency with parallel data sets. An example of such a check would be comparing data from the same volume of soil.

SECTION 4.0
FIELD QC SAMPLES

As a check on field sampling, QA/QC samples will be collected during each sampling event. Definitions for field QA/QC samples are presented below.

4.1 FIELD DUPLICATES

A field duplicate is defined as a second sample collected independently at the same sampling location during the same sampling event that produced the primary sample. While soil samples are not considered true duplicates, the results serve to indicate whether the contamination in the matrix is uniform. For XRF analyses, individual samples will be homogenized, as required, due to the heterogeneity of the material.

At least 10 percent of the total daily soil samples for off-site fixed-laboratory metals analysis will be submitted as field duplicate samples to determine the precision of the sampler and the analytical laboratory. Duplicate samples will be prepared in the same manner as other samples and will be given the sample designation "D" to indicate that it is a duplicate sample. Field duplicate samples will be analyzed for CAM-17 metals by EPA Method 6010B.

4.2 BLANKS

Equipment blanks consist of ASTM Type II water (or equivalent) poured into or pumped through the sampling device following decontamination. This blank is transferred to a sample bottle appropriate for the analysis and transported to the laboratory.

Equipment blanks will be prepared when a particular piece of sampling equipment was employed for sample collection and subsequently decontaminated in the field for use in additional sampling. The equipment blank will be taken in the field by collecting a blank water rinse from the equipment (e.g. hand auger bucket) in the appropriate pre-preserved container after execution of the last step of the field decontamination protocol. One equipment blank will be collected per team for each day of testing. Each equipment blank will be analyzed for CAM-17 metals by EPA Method 6010B.

Trip blanks are used to measure potential contamination of samples by volatile organic compounds during transport. The trip blank consists of a vial filled by the laboratory with ASTM Type II water, shipped to the field, and returned to the laboratory in a cooler that contains samples for VOC analysis. A trip blank shall be included in every cooler containing samples for VOC analysis (Method 8260); the trip blank sample will be analyzed for VOCs. A trip blank shall be included in every cooler containing samples for analyses for TPH-g (Method 8015) and analyzed for TPH-g (Method 8015). VOC and TPH analyses will not generally be performed on this project although may be required in certain instances for waste characterization or backfill sampling purposes.

SECTION 5.0
SAMPLING PROTOCOLS

Detailed soil sampling protocols are provided in the draft Workplan (Parsons, 2015a).

5.1 SAMPLE CONTAINERS

The laboratory will provide sample containers, labels, chain-of-custody forms, and coolers to the project site. Properly cleaned sample containers must be used so that no target compound contamination occurs from contact with the sample container. The laboratory will provide documentation attesting to the cleanliness of the containers following their cleaning procedures. A certificate of cleanliness will be provided for any commercially purchased sample containers.

It is equally important to use preservative reagents that are free of target analytes or other contaminants. The laboratory will provide documentation attesting to the purity and quality of the reagents being provided.

Table 2 lists the types of sample containers, sample volumes, methods of preservation, and holding times for each parameter. Field team members will ship or courier samples directly to the laboratory at the end of each sampling day, which will enable the laboratory to analyze the samples within the specified holding times.

5.2 SAMPLE CONTAINMENT, PRESERVATION, AND LABELS

Sample containers and preservatives defined in Table 2 will ensure compatibility with USEPA protocols and will minimize breakage during transportation. Sample labels will be affixed to each container to identify the sample number, collector's name, date and time of collection, location of sampling point, analyses requested, and preservatives added.

5.3 FIELD SAMPLE IDENTIFICATION

A sample numbering system will be used to identify each sample collected during field investigations, including field QC samples. The numbering system will be a tracking mechanism to allow retrieval of information about a particular location and to ensure that each sample is uniquely numbered. A listing of sample numbers will be maintained by the field team leader.

Samples will be identified first by "SS" for "Soil Sample" and "LBP" for "Lead Based Paint Sample," followed by a unique property number and a unique sample identification number. SS samples will also include the bottom depth of the sampling interval. "X" will be used to distinguish samples analyzed using a XRF hand-held device; "L" will be used to distinguish laboratory samples. The following is an example of the sampling nomenclature:

XRF soil samples

(SS - Property Name – Sample Number - Bottom Depth of Sample Interval - X)

SS-001-3-X (for 0 to 3 inches)

SS-001-6-X (for 3 to 6 inches)

SS-001-12-X (for 6 to 12 inches)

SS-001-18-X (for 12 to 18 inches)

Laboratory Soil Samples

(SS – Property Name- Sample Number – Bottom Depth of Sample Interval – L)

SS-001-3-L (for 0 to 3 inches)

SS-001-6-L (for 3 to 6 inches)

SS-001-12-L (for 6 to 12 inches)

SS-001-18-L (for 12 to 18 inches)

XRF paint samples

(LBP - Property Name – Sample Number - X)

LBP-001-1-X

XRF paint samples

LBP-001-1-L

Duplicate samples will be collected for samples submitted to the laboratory. All duplicate samples will be identified with a "D", for example, SS-001-3-LD.

5.4 SAMPLE CHAIN-OF-CUSTODY

Sample custody begins in the field at the time of collection and continues throughout the laboratory analytical process. Chain-of-custody forms will be prepared at the time of sample collection and will accompany the samples to the laboratory and through the laboratory sample processing. Chain-of-custody forms will be completed for each cooler in a shipment of samples to track the samples and provide a written record of all persons handling the samples. The following information for each sample will be documented on the chain-of-custody form:

- Unique sample identification;
- Date and time of sample collection;
- Source of sample (including name, location, and sample type);
- Designation of MS/MSD;
- Analyses required;
- Name(s) of collector(s);

- Custody transfer signatures, and dates and times of sample transfer from the field to couriers and to the laboratory; and
- Bill of lading or transported tracking number (if applicable).

Shipments will be sent by courier for daily delivery to the laboratory.

5.5 LABORATORY CUSTODY PROCEDURES

Laboratory sample custody procedures must be presented in the laboratory QAP and approved by the project manager prior to shipping any samples to the laboratory. To facilitate the documentation of sample custody, the laboratory will track the progress of sample preparation, analysis, and report preparation. Samples received by the laboratory will be checked carefully for label identification, chain-of-custody forms, and any discrepancies. The laboratory will also note and record cooler temperatures, physical damage, incomplete sample labels, incomplete paperwork, discrepancies between sample labels and paperwork, broken or leaking containers, and inappropriate caps or bottles. The laboratory will send signed facsimile copies of all chains-of-custody and sample log-in receipt forms to the field manager (FM) within 24 hours of sample receipt in the laboratory. All discrepancies and/or potential problems (e.g., lack of sample volume) will be discussed immediately with the FM.

The laboratory sample custodian will provide a report to the FM of any problems observed with any of the samples received. This report will also document the condition of samples, sample numbers received, corresponding laboratory numbers, and the estimated date for completion of analysis. Written permission must be received from the FM before sending any samples originally scheduled to be analyzed at its facility to another laboratory. Analyses will not be performed on samples whose integrity has been compromised or is suspect, without prior approval from the FM.

5.6 SAMPLE HANDLING

Laboratory sample custody will be maintained by the procedures detailed in the laboratory QAP.

- If the chain-of-custody and samples correlate, and there has been no tampering with the custody seals, the "received by laboratory" box on the chain-of-custody form will be signed and dated.
- The samples will be logged into the laboratory information management system in such a manner that tracking the status of the samples (extraction, analysis dates) can be readily accomplished.
- Water samples will be stored in a secured area at a temperature of approximately 4 ± 2 degrees Celsius (°C) for all analytical fractions except for metals. Soil samples may be stored at lower temperature (as applicable) until analyses commence. Samples must be stored in coolers separate from those used to store analytical standards, reagents, and/or QC samples.
- Volatile samples will be stored separately from other samples. A storage blank must be present in the cooler storing volatile samples and analyzed weekly at a

minimum. Results of storage blank analyses must be maintained by the QA department. Corrective action is required if analyses provide evidence of cross contamination.

- The original chain-of-custody form will accompany the laboratory report submittal and will become a permanent part of the project records.
- Data generated from the analysis of samples also must be kept under proper custody by the laboratory.

Disposal of sample containers and remaining sample material will be the responsibility of the laboratory. Samples should be disposed of appropriately when all analyses and related QA/QC work are completed (Section 15).

SECTION 6.0
FIXED-BASE LABORATORY ANALYTICAL PROCEDURES

Application of a specific analytical method depends on the sample matrix and the analytes to be identified. Methods for each of the parameters likely to be included in the analytical program, as well as detection limits, are discussed in the following subsections. All analytical methods are USEPA approved. Samples will be maintained for an extended period before disposal to allow review of data and to maintain the option of reanalysis if the results are suspect. Samples will be maintained under a laboratory internal chain of custody system, in order to retain sample integrity documentation.

6.1 ANALYTICAL METHODS

Analytical procedures will follow established USEPA method protocols. Approved methods are presented in summarized below. The referenced methods are defined in the USEPA *Test Methods for Evaluating Solid Waste, Physical and Chemical Methods, SW846, 3rd Edition, Update III* (1996). Whenever SW-846 methods are not appropriate, recognized methods from source documents published by USEPA, ASTM or other organizations with appropriate expertise will be used. While most analyses required for this project will focus on metals, particularly lead, it is anticipated that other analytical methods will be required for other project purposes (e.g., to support waste characterization or backfill sampling).

Exide Facility Off-Site Assessment and Remediation
Analytical Methods

Parameter	Analytical Method
CAM-17 Metals	EPA Method 6010B
Mercury	EPA Method 7471B
Lead	EPA Method 6010B/7000A
Hexavalent Chromium	EPA Method 7196A
Semivolatile Organic Compounds (SVOCs)	EPA Method 8270C
Polynuclear Aromatic Hydrocarbons (PAHs)	EPA Method 8310
Volatile Organic Compounds(VOCs)	EPA Method 8260B
Polychlorinated Biphenyls (PCBs)	EPA Method 8082A
Organochlorine Pesticides	EPA Method 8081B
Moisture Content	ASTM D2216
Total Petroleum Hydrocarbons – Gasoline Range Organics (GRO) (TPH-g)	EPA Method 5035/Method 8015M
Total Petroleum Hydrocarbons – Diesel Range Organics (TPH-d)	Method 8015M
Total Petroleum Hydrocarbons – Oil Range Organics (TPH-o)	Method 8015M

6.2 DETECTION AND QUANTITATION LIMITS

This section describes the terms, definitions, and formulas that will be used for detection and quantitation limits.

6.2.1 Method Detection Limit

The method detection limit (MDL) is the lowest concentration at which a specific analyte in a matrix can be measured and reported with 99-percent confidence that the analyte concentration is greater than zero. MDLs are experimentally determined and verified for each target analyte of the methods in the sampling program. Instrument-specific MDLs are analyzed in accordance with 40 CFR Part 136. The lab will spike at a level equal to the lowest calibration standard. In order to maintain reporting consistency, if multiple instruments are used for the same method, the lab will report down to the highest MDL between all instruments so that all MDLs for a given analyte are at or below the reported MDL. MDLs are verified quarterly with a spike at ½ of the low calibration standard. Since MDLs are verified quarterly and in accordance with California Department of Health Services Environmental Laboratory Accreditation Program (ELAP) policy on Method Detection Limits, annual MDL studies are not performed unless a problem is identified during the quarterly verification process. MDLs are based on the results of seven matrix spikes at two times the estimated PQL, and are statistically calculated in accordance with the Title 40, Code of Federal Regulations (CFR) Part 136 (40 CFR 136), Appendix B. The standard deviation of the seven replicates is determined and multiplied by 3.14 (i.e., the 99-percent confidence interval from the one-sided student t-test). Where practicable, MDLs must be lower than the risk-based criteria determined for the project.

The MDLs to be used are intended to allow that both non-detected and detected target compound results will be usable to the fullest extent possible for the project. An MDL check sample, an interference-free MS with all method target compounds, must be analyzed following the MDL study to determine if reasonable MDL concentrations have been achieved. The MDL check sample should be at a concentration of approximately two times the MDL. If any target compound is not recovered, the MDL study must be repeated. In this case, the repeated MDL study should be performed with a higher concentration, based on the analyst's judgment, of the target compounds which failed in the MDL check sample. The MDLs shall be verified quarterly by running a standard at ½ the concentration of the lowest standard of the initial calibration. If the verification analysis shows lack of adherence to the determined MDLs, then the MDL study shall be repeated.

6.2.2 Sample Quantitation Limit

Sample quantitation limits (SQLs) are defined as the MDL multiplied by the dilution factor (DF) required to analyze the sample, and corrected for moisture or sample size. These adjustments may be due to matrix effects or to the high concentrations of some analytes. For example, if an analyte is present at a concentration that is greater than the linear range of the analytical method, the sample must be diluted for accurate quantitation. The DF raises the reporting limit, which then becomes the SQL. Because the reported SQLs take into account sample characteristics and analytical adjustments, they are the most relevant quantitation limits for evaluating non-detected chemicals.

6.2.3 Detection Limit Goals

To define analytical data reporting limits that meet project DQOs, potential risk-based screening criteria that the DTSC has adopted for the Exide project were considered. For lead, the risk-based soil screening criteria of 80 mg/kg will serve as the primary residential soil cleanup goal. This level will be easily achieved by the indicated analytical method. However, a lower reporting limit is necessary to support the statistical evaluations that will be utilized to estimate average soil-lead concentrations at individual residential properties. Therefore, in order to characterize the potential full range of lead concentrations that may be encountered, a lead reporting limit of 1.0 mg/kg is specified.

PQLs supplied by the laboratory are summarized in Attachment 1.

6.2.4 Practical Quantitation Limit

The practical quantitation limits are the lowest matrix-specific concentrations that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. All sample results will be reported at or above the MDL for each analyte. All results above the MDL, but below the PQL, will be qualified in the data deliverable from the laboratory with a "J" flag. The "J" flag will denote the sample result as below the PQL. Where practical, MDLs must be lower than the risk-based criteria determined for the project. Laboratories must verify the PQLs by analyzing a standard at or below the PQL when performing the initial calibration curve.

Reporting limits are the lowest reported concentration provided on the sample-analysis data report, after corrections have been made for sample dilution, sample weight, and (for soils and sediments) amount of moisture in the sample. Reporting limits can be as low as the MDL or exceed the PQL, depending on the matrix effects encountered during the analysis. The reporting limit is the value that indicates whether the analytical DQOs have been achieved for that sample.

SECTION 7.0
LABORATORY QC SAMPLES AND CRITERIA

Laboratory QC data are necessary to determine the precision and accuracy of the analyses, confirm matrix interferences, and demonstrate target compound contamination of sample results. QC samples will be analyzed routinely by the analytical laboratory as part of the method QC procedures. The contract laboratory Quality Assurance Program Manual (QAPM) describes its QA system. At a minimum, the laboratory must prepare and analyze a method blank, a laboratory control sample (LCS), a laboratory sample duplicate, and a continuing calibration standard with each batch of samples. A matrix spike/matrix spike duplicate (MS/MSD) shall be analyzed with each batch, providing sufficient sample was provided to the laboratory by the sampling team. If there is not sufficient sample for MS/MSD, the laboratory will prepare and analyze the LCS in duplicate. In this manner, a measure of the precision pertaining to the specific analytical batch can be determined.

SECTION 8.0
LABORATORY DATA REVIEW, REDUCTION, AND REPORTING

The following sections describe the project minimum requirements for laboratory data review, reduction, and reporting. The laboratory through its QAP and SOPs shall specify the personnel performing each function.

Level II documentation is to be provided for all data (see Section 14).

If multiple dilutions are performed, the results of each dilution are to be reported.

8.1 REVIEW PROCEDURES FOR DEFINITIVE DATA

The laboratory review of definitive data shall be a four-step process involving an evaluation by the analyst, a peer review, an administrative review, and a QA review. A checklist to document each of the review processes will be required and must be included as part of the final data deliverable. All steps are described below.

The analyst shall review 100 percent of all definitive data prior to reporting. The establishment of method detection and control limits shall be verified. Any control limit outside the acceptable ranges specified in the analytical methods shall be identified. Any trends or problems with the data shall be evaluated. The absence of records supporting the establishment of control criteria or detection limits shall be noted and explained. Analytical batch QC, calibration check samples, initial and continuing calibrations, corrective action reports, the results of reanalysis, sample holding times, and sample preservations shall be evaluated.

Samples associated with out-of-control QC data shall be identified in the data package case narrative, and an assessment of the utility of such analytical results shall be made. The check of laboratory data completeness must be documented and will ensure that:

- All samples and analyses specified in the chain-of-custody have been processed;
- Complete records exist for each analysis and the associated QC samples; and
- Procedures specified in this QAPP have been implemented.

An analyst other than the original data processor shall be responsible for performing a peer review of all steps of the data processing. One hundred percent of all data shall be reviewed. All input parameters, calibrations, and transcriptions will be checked. All manually input, computer-processed data will be checked. Each page of checked data shall be signed and dated by the verifier.

Continuing calibrations shall be compared to the initial calibration curve to determine that the analytical system is performing within acceptable range. QC sample results (LCSs , laboratory duplicates, and MS/MSD) shall be compared against stated acceptance criteria for accuracy and precision. QC data must meet acceptance levels prior to processing the analytical data. If QC standards are not met, the cause shall be determined. If the cause can be corrected without affecting the integrity of the analytical data, processing of the data shall proceed. If the resolution jeopardizes the integrity of the data, reanalysis shall be performed, if still within holding time. If the holding time

will be exceeded, the decision regarding reanalysis will be made upon conferring with the Parsons PM or designee.

An administrative review will be performed by the laboratory project manager on each data deliverable package. The review will ensure that all requirements of the laboratory and the data deliverables have been met and are complete.

A review of at least 10 percent of all data deliverable packages by a laboratory QA officer must take place prior to the administrative review and final release of the data deliverable. The data packages will be randomly selected for review.

8.2 LABORATORY DATA REPORTING FLAGS

The following qualifiers must be used by the laboratory when reporting sample results.

Qualifier	Description
J	The analyte was positively identified, the quantitation is an estimation, and/or the analyte was positively identified but the associated numerical value is greater than the SQL but less than the PQL.
U	The analyte was analyzed for, but not detected. The associated numerical value is at or below the MDL.
B	The analyte was found in an associated blank, as well as in the sample.

8.3 ASSESSMENT OF DATA USABILITY

The Project Chemist will assess data usability and apply data qualifiers to the analytical results based on adherence to method protocols and laboratory-specific QA/QC limits.

8.3.1 Data Usability Assessment

The laboratory deliverable will include the following information:

- Case narratives;
- Chain-of-custody forms;
- Summary of results by sample;
- Holding times;
- Sample temperatures during shipping and storage;
- Summary of QC results (method blanks, equipment blanks, laboratory duplicates, LCS, MS/MSD, etc.); and
- Surrogate spikes recoveries.

Data qualifiers are applied to analytical results during the data usability assessment, based on adherence to method protocols and QA/QC limits.

The validation guidelines are defined in Tables 3 and 4 and were developed in accordance with the *Superfund Methods* (USEPA, 2005) and *National Functional Guidelines for Inorganic Superfund Data Review* (USEPA, 2013). Expanded criteria for the data usability guidelines were developed where professional judgment is

recommended within the USEPA guidelines. QC guidelines are those specified in the analytical method protocols.

8.3.2 Data Reporting Qualifiers

The following definitions provide explanations of the USEPA qualifiers to be assigned to analytical results during data validation, as defined in Tables 3 and 4. The data qualifiers described are applied to sample results.

Qualifier	Description
U	The analyte was analyzed for and is not present above the reported sample quantitation limit.
J	The analyte was analyzed for and was positively identified, but the associated numerical value may not be consistent with the amount actually present in the environmental sample. The data should be considered as a basis for decision making and are usable for many purposes.
R	The data are rejected as unusable for all purposes. The analyte was analyzed for, but the presence or absence of the analyte was not verified. Resampling and reanalysis are necessary to confirm the presence or absence of the analyte.
UJ	The analyte analyzed for was not present above the reported sample quantitation limit. The associated numerical value may not accurately or precisely represent the concentration necessary to detect the analyte in the sample.

8.3.3 Assessment of Usability

Data usability will be assessed by the project chemist based on data evaluation results to determine the project PARCCs. Targeted data validation and evaluation will be performed on any result that appears to be unusual or outside the expected range. Any limitations on data use will be expressed quantitatively to the extent practicable. The outcome of this data review will be a data set appropriate to support project-specific DQOs. A DQA will be written, summarizing the findings of the data review, and providing an assessment of overall data quality and usability.

SECTION 9.0
QA REPORTS

At intervals recommended by DTSC, beginning with the initiation of sampling activities, the laboratory will submit an internal QA report that documents laboratory-related QA/QC issues to the contractor's project manager. These reports will include discussions of any conditions adverse or potentially adverse to quality, such as:

- Responses to the findings of any internal or external systems or performance laboratory audits;
- Any laboratory or sample conditions that necessitate a departure from the methods or procedures specified in this QAPP;
- Any missed holding times or problems with laboratory QC acceptance criteria; and
- The associated corrective actions taken.

Submittal of QA reports will not preclude earlier contractor notification of such problems when timely notice can reduce the loss or potential loss of quality, time, effort, or expense. Appropriate steps will be taken to correct any QA/QC concerns as they are identified. The QA reports and a summary of the laboratory QA/QC program and results will be included in the final project report.

SECTION 10.0
CORRECTIVE ACTION

The following procedures have been established to assure that conditions adverse to data quality are promptly investigated, evaluated, and corrected. Adverse conditions may include malfunctions, deficiencies, deviations, and errors.

When a significant condition adverse to data quality is noted at the laboratory, the cause of the condition will be determined, and corrective action will be taken to prevent repetition. Condition identification, cause, reference documents, and corrective action planned will be documented and reported to the contractor QA officer by the laboratory QC coordinator. Following implementation of corrective action, the laboratory QC coordinator will report the actions taken and their results to the contractor project manager and QA officer. A record of the action taken and results will be attached to the data report package. If samples are reanalyzed, the assessment procedures will be repeated, and the control limits will be reevaluated to ascertain if corrective actions have been successful.

Implementation of corrective action is verified by documented follow-up action. All project personnel have the responsibility, as part of the normal work duties, to identify, report, and solicit approval of corrective actions for conditions adverse to data quality.

Corrective actions will be initiated in the following instances:

- When predetermined acceptance criteria are not attained (objectives for precision, accuracy, and completeness);
- When the prescribed procedure or any data compiled are faulty;
- When equipment or instrumentation is determined to be faulty;
- When the traceability of samples, standards, or analysis results is questionable;
- When QA requirements have been violated;
- When designated approvals have been circumvented;
- As a result of systems or performance audits;
- As a result of regular management assessments;
- As a result of intra-laboratory or inter-laboratory comparison studies; and
- At any other instance of conditions significantly adverse to quality.

Laboratory project management and staff, such as QA auditors, document and sample control personnel, and laboratory groups, will monitor work performance in the normal course of daily responsibilities.

The laboratory QC coordinator or designated alternate will audit work at the laboratory. Items, activities, or documents ascertained to be compliant with QA requirements will be documented, and corrective actions will be mandated in the audit report. The contractor

QA officer and laboratory QC coordinator will log, maintain, and control the audit findings.

The contractor QA officer and laboratory QC coordinators are responsible for documenting all out-of-control events or non-conformance with QA protocols. A nonconformance report will summarize each nonconformance condition. The laboratory will notify the contractor project manager or QA officer of any laboratory QA/QC non-conformance issues upon their discovery. Copies of all field change requests and corrective action forms will be maintained in the project files. A stop-work order may be initiated by the contractor if corrective actions are insufficient.

SECTION 11.0
AUDITS

This section describes participation in external and internal systems audits.

11.1 SYSTEM AUDITS

System audits review laboratory operations and the resulting documentation. An onsite audit ensures that the laboratory has all the personnel, equipment, and internal SOPs needed for performance of contract requirements in place and operating. The system audits ensure that proper analysis documentation procedures are followed, that routine laboratory QC samples are analyzed, and that any non-conformance issues are identified and resolved.

11.1.1 Internal Audits

The laboratory must conduct internal system audits on a periodic basis. The results of these audits will be documented by the Laboratory QA Officer, and the laboratory will provide the Project Chemist and Task Manager with the results of these internal audits.

11.1.2 External Audits

The Project QA Officer or Task Manager may conduct an external on-site system audit of the laboratory prior to the analysis of project samples. This audit would evaluate the capabilities and performance of laboratory personnel, equipment, and procedures. It also documents the measurement systems and identifies deficiencies to be corrected by the laboratory. The QA Manager acts on audit results by documenting deficiencies and informing the Task Manager of the need for corrective action. The Task Manager may suspend operations until problems are resolved. If conditions adverse to quality are detected, or if the Task Manager requests additional audits, additional unscheduled audits may be performed.

In addition to this audit of the laboratory, various local, state and/or federal agencies may conduct an audit prior to the commencement of the project, and/or may conduct audits as deemed necessary during project execution. The frequency and schedule of any such audits will be established by the auditing agency and coordinated directly with the laboratory.

11.2 PERFORMANCE AUDITS

Laboratory performance audits may be conducted to determine the accuracy and implementation of the QAPP by the Project QA Officer or designee at any time during field sampling and analysis. Unplanned audits may be implemented if requested by the PM. The Project QA Officer will act to correct any laboratory performance problems.

SECTION 12.0
PREVENTIVE MAINTENANCE

All instrumentation shall be maintained in a manner that produces consistent, quality data and that prevents possible limitations on analytical capacity in the laboratory.

12.1 PROCEDURES

Equipment, instruments, tools, gauges, and other items requiring preventive maintenance will be serviced in accordance with the manufacturers' specified recommendations and written procedures developed by the operators.

12.2 SCHEDULES

Manufacturers' procedures identify the schedule for servicing critical items to minimize downtime of the measurement system. It will be the responsibility of the individual operator assigned to a specific instrument to adhere to the instrument maintenance schedule and to promptly arrange any necessary service. Servicing of the equipment, instruments, tools, gauges, and other items will be performed by qualified personnel.

The laboratory will establish logs to record maintenance and service procedures and schedules. All maintenance records will be documented and will be traceable to the specific equipment, instruments, tools, and gauges. Records produced for laboratory instruments will be reviewed, maintained, and filed by the operators at the laboratories.

12.3 SPARE PARTS

A list of critical spare parts will be requested from manufacturers and identified by the operator. These spare parts will be stored for availability and use in order to reduce downtime due to equipment failure and repair.

SECTION 13.0
SECURITY

All access to the laboratory must be secured and controlled. The laboratory must have controlled access to sample storage and data handling areas. All computer systems must be electronically secured with a system of write access that can be fully documented with an audit trail. All laboratory visitors must sign in and out of the building and be escorted while on site.

SECTION 14.0
DATA DELIVERABLES

The deliverables required for this project are in both hard-copy and electronic format. These formats are described below.

14.1 HARDCOPY DATA DELIVERABLES

Level II data packages are required from the off-site fixed laboratory. The laboratory will be expected to provide Level II packages within 10 workings days from the time of receipt of samples unless otherwise specified on the COCs.

14.2 ELECTRONIC DATA DELIVERABLES

To facilitate data handling and management, laboratory data will be provided to Parsons in an electronic format. All data contained in the electronic data files will correspond identically to the data contained in the original laboratory reports and other documents associated with sampling and the laboratory hardcopy data deliverable packages. The format of the electronic data deliverable will be arranged between the Parsons data manager and the laboratory data management personnel.

SECTION 15.0
FINAL SAMPLE DISPOSITION

Upon completion of all required analyses and acceptance of the data reported, the laboratory will be responsible for proper disposal of any remaining samples, sample containers, shipping containers, and Styrofoam™ or plastic packing materials in accordance with sound environmental practice, based on the sample analytical results. Unused samples and containers found to be nonhazardous generally will be disposed after 180 days following completion of the analysis. In cases where the data package meets the project QA/QC requirements and no apparent anomalies are present in the data set, the Project Chemist may authorize the laboratory to dispose of the samples at an earlier date. The laboratory shall maintain proper records of waste disposal and shall have disposal company contracts on file for inspection.

All raw and processed data generated during the analysis of project samples must be stored for a period of five years. Revised copies of the applicable SOPs and QAPs must also be maintained and available should the data be required. Should the laboratory go out of business, all original records related to project samples shall be provided to project personnel.

SECTION 16.0

SUBCONTRACT LABORATORY SERVICES OTHER THAN THE PRIME LABORATORY

The laboratory will assume responsibility for providing all analytical services specified in the laboratory subcontracting agreement. Should it be agreed in writing that the laboratory may use an additional subcontract laboratory facility, the primary laboratory will supply to the Task Manager the SOPs, MDL studies, and QA plans from the other laboratory that is used. The laboratory will be responsible for communicating all analytical guidelines and QC requirements of the project to this laboratory. Both QA Officers will monitor the data from each subcontract laboratory and correct any QC nonconformance.

SECTION 17.0
REFERENCES

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TABLES

TABLE 1
DATA QUALITY OBJECTIVES (DQOs) FOR OFF-SITE SOIL SAMPLING AND CLEANUP
USEPA'S (2000) SEVEN-STEP SYSTEMATIC PLANNING PROCESS

EXIDE FACILITY
VERNON, CALIFORNIA

Step 1: Step 1: State the Problem • Describe the problem and develop a conceptual site model (CSM) • Identify planning team members and decision makers.		• To determine if aerially deposited heavy metals including lead have impacted off-site residential soils to the extent that such concentrations are a health hazard to the residents living in the Subject Area. • Based on the sampling results of the Northern and Southern Initial Assessment Areas [also referred to collectively herein as the Initial Assessment Areas(s)], elevated lead was detected at 146 residential properties already sampled in the Subject Area, which suggest that potentially thousands of residential properties may be affected. As a result, additional sampling to characterize the extent of lead in soil and subsequent soil removal is required on the properties with the greatest potential for exposure. • Based on the conceptual site model (CSM), potential sources of lead in soils include aerially deposited particulate emissions from Exide, from other local/regional historical sources including fuel combustion emissions, and from lead-based paint that was historically used on these residential structures. The primary exposure pathways of concern for soil are incidental ingestion, dermal contact, and inhalation of particulates from soil and indoor dust. • Planning team members include staff from Cal/EPA's Department of Toxic Substances and Control (DTSC) Hazardous Waste Management Program, Public Participation, and Human and Ecological Risk Office (HERO), the Exide Technologies Advisory Group, and Parsons.
Step 2: Step 2: Identify the Decision • Identify the principal study question(s) • Define alternative actions • Define a decision statement		• The principal study question is to determine if soil concentrations at individual properties exceed the site-specific soil screening level for lead of 80 mg/kg, which is protective of incidental ingestion, dermal contact, and inhalation of particulates. • Sampling will be conducted at residential properties located within the Subject Areas. DTSC estimates that a total of 15 to 17 individual soil sample locations and up to 10 sample locations on structure exteriors. Soil samples from 0 to 3 inches will be screened using an X-Ray fluorescence (XRF) analyzer, similar or superior to the Niton Xli700 Series, at 15 to 17 samples per property. Based on the XRF results, if any 0 to 3-inch samples exceeds a concentration for lead of 80 mg/kg with a margin of error of ± 30 Percent (± 30 percent of the element limit; currently the lead limit is 80 mg/kg, therefore, a threshold of <56 mg/kg is compliant, >104 mg/kg is non-compliant, and 56 to 104 mg/kg is inconclusive). The margin of error of ± 30 percent may be different for other XRF equipment and may need to be adjusted. Laboratory confirmation samples of XRF screening results will be collected at a rate of 10 percent (i.e., minimum of two samples per property) and submitted to an off-site fixed laboratory for CAM 17 metals analysis. • Dripline samples will be collected from each residential property. In addition, presuming that downspouts are encountered, soil samples from at least one location will be collected from below any downspouts identified on the property.

Step 3: Step 3: Identify the Decision Inputs • Identify the information needed and the corresponding sources • Define the basis for determining the action levels • Identify sampling and analysis methods that can meet the data requirements		• This QAPP and the associated Workplan includes the analytical methods to be used and the screening levels for each parameter being tested to obtain the desired cleanup or research objective as determined by DTSC. An approved environmental laboratory that is California ELAP-certified will prepare the samples and perform the analysis. • The proposed soil investigation will be conducted in accordance with pertinent DTSC and USEPA guidance. This includes, but is not limited to sample collection and analysis, decontamination procedures, and other QA/QC testing. • The soil lead screening level of 80 mg/kg was developed by the DTSC and is protective of incidental ingestion, dermal contact, and inhalation of particulates.
Step 4: Step 4: Define the Boundaries of the Study • Define the target investigative area • Specify the spatial boundaries for the investigation • Determine the time frame for collecting the data • Determine the practical constraints on collecting the data • Determine the smallest unit for which decisions will be made		• The areas subject to the investigation are designated as portions of the incorporated and/or unincorporated cities of Maywood, Boyle Heights, East Los Angeles, Commerce, Bell, and Huntington Park. Only properties with residential levels of exposure to lead will be addressed under this Workplan. • The spatial boundaries of the target investigative area are shown in the Workplan. • The collection and review of soil data for the initial 1,000 residential properties will occur through May 2016, with the soil sampling data being submitted to DTSC as soon as they become available. This proposed schedule is contingent upon no delays in the collection and analysis of samples due to factors such as the weather (e.g., rain) or access agreements. • The sampling approach utilizes XRF field analyses, which allows for a rapid turnaround time and large number of soil samples to be screened for the presence of elevated lead. Use of the XRF requires a representative soil sample to be prepared for the analysis. Parsons and DTSC will work to ascertain the optimum sample preparation approach that provides cost-effective technically defensible data. The field results will be confirmed through confirmatory sampling analyzed by an off-site fixed laboratory using more refined sample preparation and analytical methods. • The smallest "decision unit" will correspond to one residential property (i.e., each property is a separate decision unit).
Step 5: Step 5: Develop Decision Rules • Specify an appropriate decision unit		Given the practical limitations associated with the amount of data likely to be available within a decision unit, soil results from individual samples within the decision unit will be used (i.e., maximum detected concentrations within the decision unit). However, if adequate data are available, the appropriateness of using an upper confidence level (UCL) estimate of the mean concentration will be discussed with the team. Laboratory and XRF results (including the margin of error as discussed under Step 2), will be compared to the following

	parameter (e.g., maximum or average soil-gas concentration) • Confirm that the analytical detection limits are less than the Action Levels • Develop decision rules (If…then… statements)	Decision Rules: • If discrete sample results identify soil lead concentrations greater than or equal to 1,000 mg/kg in any single soil sample in the uppermost depth interval (0 to 3 inches) or in two or more soil samples at any depth, then the soil removal for that property will be given an initial *Priority 1* status. • If discrete sample results identify lead concentrations greater than 400 mg/kg in any single soil sample in the uppermost depth interval (0 to 3 inches) or in two or more soil samples at any depth, then the soil removal will be given a *Priority 2* status. However, if these properties are occupied by children (under 7 years) or pregnant women and bare soils are present, then soil removal may be given a *Priority 1* status. • If the soil-lead concentration is greater than or equal to 80 mg/kg based on a property-wide 95% upper confidence limit (UCL) of the mean for the upper-most depth interval (0 to 3 inches), then the property will be given a *Priority 3* status, unless occupants include children (under 7 years) or pregnant women, or bare soils are present. If such conditions are present on a property, then soil removal will be given a *Priority 2* status.
Step 6: Step 6: Specify Tolerable Limits on Decision Errors		Sample data are subject to random and systematic errors during field collection and sample analysis. The combination of errors is referred to as "total study error." The two contributors to the total study error are the statistical Sampling Design Error and the Measurement Error (USEPA, 2000a): • Sampling Design Error, which is influenced by inherent variability over space and time, sample collection, and the number of samples. • Measurement Error, which is influenced by random and systematic errors introduced during sample preparation, sample analysis, data reduction, transmission, and storage. Since the total study error directly affects the possibility of making a decision error, the total decision error must be managed by minimizing the sample design and measurement errors. To minimize the sampling error, the following procedures will be employed: • Utilize discrete sampling procedures to obtain representative samples; • Obtain a sufficient number of samples for robust statistical analysis (samples from approximately 15 different locations per residential property); and • Collect samples from bare exposed soil that have not been recently disturbed or open grassed areas away from structures or tall trees. To minimize measurement error, the following procedures will be utilized: • Locate measurement points (sample locations) where maximum deposition is predicted; • Collect the samples in a standardized manner; • Label each sample and transport it to the laboratory under chain-of-custody; • Specify that the laboratory use accepted USEPA Methods and report the data using the proper unit; • Specify that the laboratory participates in regular performance testing, is certified in California, and has ELAP certification; • Receive the analytical data from the laboratory in an electronic format to minimize transcription errors, and • Perform a quantitative and qualitative review (data validation) of the analytical data to verify the reliability of the data.

Step 7:	**Step 7: Optimize the Design for Obtaining Data**	Data variability may have an effect on the sampling design. Upon review of the analytical data, the sampling frequency, sampling locations, sample preparation procedures, and/or the number of samples analyzed may be changed to optimize the design. The design options will be evaluated based on cost and the ability to meet the DQOs.

Table 2

Requirements for Containers, Preservation Techniques, Sample Volumes, and Holding Times
Exide Facility Off-Site Soil Investigation and Cleanup

Name	Analytical Methods	Matrix	Container [a/]	Preservation [b/]	Minimum Sample Volume or Weight	Maximum Holding Time
Chloride, Nitrate, Sulfate	SW9056 or E300.0	Water	P, G	4°C	50 ml	28 days for Cl^-, and SO_4^{-2}; 48 hours for NO_3^-
Total Organic Carbon	SW9060 or E415.1	Water	G, Teflon® - lined cap	HCl to pH <2, 4°C	500 ml	28 days
		Soil	T, G Teflon® - lined cap	4 °C	8 ounces	28 days
Metals [c/]	SW6010B, SW6020 and SW7470A	Water	P	HNO_3 to pH <2	500 ml	180 days
	Soil		T, G	None	8 ounces	180 days
Chlorinated Herbicides	SW8151A	Water	GA, Teflon®-lined cap	4 °C	2 liters	7 days until extraction and 40 days after extraction
		Soil	T, G, Teflon® - lined cap	4 °C	8 ounces	14 days until extraction and 40 days after extraction
Volatile Organic Compounds	SW8260B	Water	G, Teflon®-lined septum	4 °C, HCl to pH < 2, 0.008% $Na_2S_2O_3$ [d/]	3 x 40 ml	14 days; 7 days if unpreserved by acid
		Soil	EnCore□ Sampler	4°C or preservation by methanol or by freezing	3x EnCore□ Sampler	48 hours at 4 °C, 7 days in methanol or frozen
Total Petroleum Hydrocarbons	SW8015	Water	G, Teflon®-lined septum	°C, HCl to pH < 2, 0.008% $Na_2S_2O_3$ [d/]	3 x 40 ml	14 days; 7 days if unpreserved by acid
Organochlorine Pesticides	SW8081A	Water	GA, Teflon®-lined cap	°C	2 liters	7 days until extraction and 40 days after extraction
		Soil	T, G, Teflon® - lined cap	°C	8 ounces	14 days until extraction and 40 days after extraction
Semi-Volatile Organic Compounds	SW8270C	Water	GA, Teflon®-lined cap	°C, 0.008% $Na_2S_2O_3$ [d/]	2 liters	7 days until extraction and 40 days after extraction
		Soil	T, G w/ Teflon® - lined cap	4°C	8 ounces	14 days until extraction and 40 days after extraction
Polychlorinated Biphenyls (PCBs)	SW8082	Water	GA, Teflon®-lined cap	°C, 0.008% $Na_2S_2O_3$ [d/]	2 liters	7 days until extraction and 40 days after extraction
		Soil	T, G w/ Teflon® - lined cap	4°C	8 ounces	14 days until extraction and 40 days after extraction

Table 2

Requirements for Containers, Preservation Techniques, Sample Volumes, and Holding Times
Exide Facility Off-Site Soil Investigation and Cleanup

Name	Analytical Methods	Matrix	Container [a/]	Preservation [b/]	Minimum Sample Volume or Weight	Maximum Holding Time
Polychlorinated Biphenyls (PCBs)	EPA 1668A	Water	GA, Teflon®-lined cap 4	°C, 0.008% $Na_2S_2O_3$ [d/]	2 liters	7 days until extraction and 40 days after extraction
		Soil	T, G w/ Teflon® - lined cap	4°C	8 ounces	14 days until extraction and 40 days after extraction

a/ Polyethylene (P); glass (G); glass amber (GA), brass or stainless steel sleeves in the sample barrel (T).

b/ No pH adjustment for soil.

c/ All metals collected for a dissolved portion analysis will be filtered in the field prior to preservation.

d/ Preservation with 0.008 percent $Na_2S_2O_3$ is only required when residual chlorine is present.

Table 3

Flagging Conventions for Data Evaluation and Validation of Organic Methods
Exide Facility Off-Site Soil Investigation and Cleanup

Quality Control Check	Evaluation	Flag	Samples Affected
Holding Time	Holding time exceeded for extraction or analysis by > 2 times Holding time exceeded for extraction or analyses by < 2 times	J positive results R non-detects J positive results UJ non-detects	Sample, MS/MSD [a/] Sample, MS/MSD
Sample Preservation	Sample not preserved	J positive results UJ non-detects	Sample, MS/MSD
Temperature Blank	>8°C >20°C (Volatile Compounds)	J positive results (except PCBs) UJ non-detects (except PCBs) R all results	All samples in same cooler All samples in same cooler
Tune	Ion abundance criteria	J positive results UJ non-detect results	All associated samples in analysis batch
	Set critical ions as defined in the NFG [b/]	R all positive results and non-detects	All associated samples in analysis batch
Initial Calibration (ICAL)	GC/MS: [c/] RRF [d/] <0.05	R non-detects J positive results	Compound in all associated samples in analysis batch
	%RSD ≥30% and all initial calibration RRF ≥0.05	UJ non-detects J positive results	Compound in all associated samples in analysis batch
Initial Calibration (ICAL) (continued)	If %RSD >2X control criteria	R all positive results and non-detects	Compound in all associated samples in analysis batch
	GC: [e/] For multi-component target compounds, at least 3 peaks used with a RT [f/] window of ±0.07 minutes each	J positive results UJ non-detects	Compound in all associated samples in analysis batch
	%RSD [g/] linearity: Correlation coefficient of curve < 0.995 but > 0.990 Correlation coefficient of curve < 0.990 %RSD > 20% If %RSD >2X control limit	J positive results UJ non-detects R all positive and non-detects J positive results UJ non-detects R all positive and non-detects	Compound in all associated samples in analysis batch Compound in all associated samples in analysis batch

Table 3

Flagging Conventions for Data Evaluation and Validation of Organic Methods
Exide Facility Off-Site Soil Investigation and Cleanup

Quality Control Check	Evaluation	Flag	Samples Affected
Calibration Verification (CCAL)	GC/MS: %D [h/] ≥25% and RRF≥0.05	J positive results UJ non-detects	Compound in all associated samples in analysis batch
Calibration Verification (CCAL) (Continued)	If %D is >2X control criteria	R all positive results and non-detects	Compound in all associated samples in analysis batch
	RRF <0.05	J positive results R non-detects	Compound in all associated samples in analysis batch
	GC: Correlation coefficient of curve < 0.995 but > 0.990 Correlation coefficient of curve < 0.990 %D >15%	J positive results UJ non-detects R all positive and non-detects J positive results UJ non-detects	Compound in all associated samples in analysis batch Compound in all associated samples in analysis batch
	If %D is > 2X control criteria	R all positive results and non-detects	Compound in all associated samples in analysis batch
Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD)	LCS or LCSD single compound: %R <30%	R all positive results and non-detects	Spiked compound only in all associated samples.
	%R >UCL [1/] but < 150%.	J positive results No qualification for non-detects	Spiked compound only in all associated samples.
	% R ≥ 30% but < LCL [j/]	J positive results UJ non-detects	Spiked compound only in all associated samples.
	% R >UCL and >150%	R all positive results good/non-detects	Spiked compound only in all associated samples.
	If ≥ 50% of all LCS or LCSD spiked compounds are out of control: RPD [k/] >control limit	R all positive results and non-detects J positive results	All detected spike compounds in all samples All detected spike compounds in all samples

Table 3

Flagging Conventions for Data Evaluation and Validation of Organic Methods
Exide Facility Off-Site Soil Investigation and Cleanup

Quality Control Check	Evaluation	Flag	Samples Affected
Method Blank	Multiply value by 5, common lab contaminants multiply by 10 [l/]	U flag reported results < calculated value	All samples in extraction batch
Equipment Blank	Convert to soil units, if applicable, multiply by 5, common lab contaminants multiply by 10 [b/]	U flag reported results < calculated value	All samples, same field team, matrix and date (water) or all samples, same field team, matrix (soil)
Trip Blank	Convert to soil units, if applicable, multiply by 5, common lab contaminants multiply by 10 [b/]	U flag reported results < calculated value	All volatile samples shipped in the same cooler
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	MS or MSD single compound: %R <10%	R all positive results and non-detects	Affected compound in native sample MS/MSD
	%R >UCL but < 200%	J positive results No qualification for non-detects	Affected compound in native sample MS/MSD
	% R [m/] ≥ 10% but < LCL	J positive results UJ non-detects	Affected compound in native sample MS/MSD
	% R >UCL and >200%	R all positive results and non-detects Al	compounds in native sample
	If ≥ 50% of all MS or MSD spiked compounds are out of control:	R all positive results and non-detects Al	compounds in native sample
	When sample conc. is >4X spike conc.	No evaluation required	None
	RPD > control limit	J positive results No qualification for non-detects	Affected compound in native sample MS/MSD
Surrogates GC/MS SEMI-VOA	If 2 or more surrogates from the same chemical family group: %R > UCL	J positive results	All associated compounds in sample
	%R < LCL and ≥ 10%	J positive results UJ non-detects	All associated compounds in sample
	Any one < 10%	J positive results R non-detects	All associated compounds in sample
GC Methods [n/] and GC/MS VOA	%R > UCL	J positive results	All compounds in associated sample
	%R < LCL and ≥ 10%	J positive results UJ non-detects	All compounds in associated sample
	%R < 10%	J positive results	All compounds in associated sample

Table 3

Flagging Conventions for Data Evaluation and Validation of Organic Methods
Exide Facility Off-Site Soil Investigation and Cleanup

Quality Control Check	Evaluation	Flag	Samples Affected
		R non-detects	
Internal Standards (IS)	RT Difference > 30 seconds between sample and 12-hour standard	R all positive results and non-detects Al	associated compounds in sample
(GC/MS)	IS extracted ion area counts <50% \of last CCAL	J positive results UJ non-detects	All compounds in associated sample
	IS extracted ion area counts >200% of last CCAL	J positive results	All compounds in associated sample
Retention Time Windows (RTW)	Analyte peak not within RTW	Report positive result as non-detect, (professional judgment should be used prior to eliminating detections)	All affected compounds
Second-Column Confirmation	Primary and confirmation results do not agree within a factor of 50 percent.	J positive results	All affected compounds
Field Duplicates	RPD > 35% water or soil	Discuss impacts in data quality assessment report	Field duplicate pair
Breakdown check (DDT) (SW8080A)	% Breakdown for DDT > 20%	J positive DDT, DDE, and DDE results R non-detects for DDT if DDD and DDE are positive	Samples following the last in control standard
Breakdown check (Endrin) (SW8080A)	%Breakdown for Endrin > 20%	J positive endrin, endrin aldehyde, and endrin ketone results R non-detects for endrin if endrin aldehyde and endrin ketone are positive	Samples following the last in control standard

a/ MS/MSD = Matrix spike/matrix spike duplicate. k/ RPD = Relative percent difference.
b/ NFG = National Functional Guidelines. l/ Common lab contaminants: methylene chloride, acetone, 2-butanone, and phthalates.
e/ GC/MS = Gas chromatograph/mass spectroscopy m/ %R = Percent recovery.
d/ RRF = Relative response factor. n/ Number of surrogates varies with method. Pesticides and PCBs are surrogate specific
e/ GC = Gas chromatography and are evaluated as independent chemical family groups.
f/ RT = Retention time. o CCAL = Continuing calibration
s/ RSD = Relative standard deviation

h/ %D = Percent difference.
i/ UCL = Upper control limit.
j LCL = Lower control limit.

Table 4

Flagging Conventions for Data Evaluation and Validation of Inorganic and Wet Chemistry Methods
Exide Facility Off-Site Soil Investigation and Cleanup

Quality Control Check	Evaluation	Flag	Samples Affected
Holding Time	Holding time exceeded for digestion or analysis by < 2 times exceeded by > 2 times	J positive results UJ non-detected results J positive results R non-detects.	Sample only Sample only
Sample Preservation	Sample preservation requirements not met	J positive results UJ non-detects for all methods except mercury R mercury non-detects	Sample only
Temperature Blank	>8°C	J positive results UJ non-detects	Samples in same cooler
Initial (Multipoint) Calibration	Correlation coefficient of curve < 0.995 but > 0.990 Correlation coefficient of curve < 0.990	J positive results UJ non-detects R positive results R non-detects	All associated samples in analysis batch All associated samples in analysis batch
Calibration Standard Check	Recovery above UCL [a/] or below LCL [b/]	R positive results R non-detects	All associated samples in analysis batch
Calibration Verification: ICV [c/], CCV [d/]	ICP/GFAA, WET Chemistry: %R [e/] between 75-89% or 111-125%	 UJ non-detects J positive results No qualification for non-detects with 111-125%R	 All associated samples in analysis batch for ICV
	%R < 75%	R positive results	Samples after failed CCV until next in control CCV
	%R > 125%	R positive results No qualification for non-detects	Samples after failed CCV until next in control CCV
	Hg: %R between 65-79% or 121-135% %R between 65-79% %R < 65%	J positive results UJ non-detects R positive results	Samples after failed CCV until next in control CCV All associated samples in analysis batch

Table 4

Flagging Conventions for Data Evaluation and Validation of Inorganic and Wet Chemistry Methods
Exide Facility Off-Site Soil Investigation and Cleanup

Quality Control Check	Evaluation	Flag	Samples Affected
	%R > 135%	R positive No qualification for non-detects	
Interference Check Sample (ICS) ICS Continued (ICP Only)	%R > UCL %R between 50-79% %R < 50%	J positive results No qualification for non-detects J positive results UJ non-detects R positive results No qualification for non-detects	All associated samples in analysis batch
Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD)	LCS or LCSD single analyte: %R <30% [f/]	R all positive results and non-detects	Spiked compound only in all associated samples.
	%R >UCL but < 150%	J positive results No qualification for non-detects	Spiked compound only in all associated samples.
	% R ≥ 30% but < LCL	J positive results UJ non-detects	Spiked compound only in all associated samples.
	% R >UCL and >150%	R all positive results and non-detects	Spiked compound only in all associated samples.
	If ≥ 50% of all LCS or LCSD spiked compounds are out of control:	R all positive results and non-detects	All compounds in all associated samples
	RPD [g/] > control limit	J positive results No qualification for non-detects	All detected spike compounds in all samples
Blanks: MB [h/], ICB [i/], CCB [j/]	If the absolute value of the blank is >MDL, then multiply value by 5, convert to soil units if applicable	U flag reported results < calculated values	All samples in digestion batch (MB) All samples in analysis batch (ICB, CCB)
Equipment Blank	If the absolute value of the blank is >MDL, then multiply value by 5, convert to soil units if applicable	U flag reported results < calculated values	All samples, same field team, matrix and date (water) or all samples, same field team, matrix (soil)

Table 4

Flagging Conventions for Data Evaluation and Validation of Inorganic and Wet Chemistry Methods
Exide Facility Off-Site Soil Investigation and Cleanup

Quality Control Check	Evaluation	Flag	Samples Affected
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	MS or MSD single compound:		
	%R <10%	R all positive results and non-detects	Affected compound in native sample MS/MSD
	%R >UCL but < 200%	J positive results No qualification for non-detects	Affected compound in native sample MS/MSD
	% R ≥ 10% but < LCL	J positive results UJ non-detects	Affected compound in native sample MS/MSD
	% R >UCL and >200%	R all positive results and non-detects	All compounds in native sample
	If ≥ 50% of all MS or MSD spiked compounds are out of control:	R all positive results and non-detects	All compounds in native sample
	When sample conc. is <4X spike conc.	No evaluation required	None
	RPD > control limit	J-positive results No qualification for non-detects	Affected compound in native sample MS/MSD
Serial Dilution (ICP Only)	If concentration is > 50 times MDL and % difference > control limit	J positive results UJ non-detects	All samples in digestion batch if analytical spike not performed
MSA [k]	MSA not done	J positive results No qualification for non-detects	Sample only
	MSA spike levels inappropriate	J positive results No qualification for non-detects	Sample only
	r ≤0.995	J positive results No qualification for non-detects	Sample only
Field duplicates	RPD > 35% water or soil	Discuss in data quality assessment report	Field duplicate pair

a/ UCL = Upper control limit.
b/ LCL = Lower control limit
c/ ICV = Initial calibration verification.
d/ CCV = Continuing calibration verification.
e/ %R = Percent recovery.
f/ Exceptions occur when the historical control limits are below or above the maximum/minimum %R value. When this occurs, the historical control limit takes precedence. Data are qualified as unusable only after the historical control limit is exceeded.

g/ RPD = Relative percent difference.
h/ MB = Method blank.
i/ ICB = Initial calibration blank.
j/ CCB = Continuing calibration blank.
k/ MSA = Method of standard addition.

ATTACHMENT 1

LABORATORY PQLs

Mr. Mark Noorani
Orange Coast Analytical, Inc.
3002 Dow Ave Ste 532
Tustin, CA, 92780

Lab Reference #: OCA 15000
Project Name: OU3
Project #:

Metals

Client Sample ID	Lab Sample Number	Date Received	Date Sampled	Matrix
Example Sample	15000-001	8/12/2014	8/12/2014	Soil

ANALYTE	EPA Method	Result	Units	Date Extracted	Date Analyzed	Qual	DF
Antimony	6010B	<5	mg/kg		08/24/14	--	1
Arsenic	6010B	<1	mg/kg		08/24/14	--	1
Barium	6010B	<0.5	mg/kg		08/24/14	--	1
Beryllium	6010B	<0.5	mg/kg		08/24/14	--	1
Cadmium	6010B	<0.5	mg/kg		08/24/14	--	1
Chromium	6010B	<0.5	mg/kg		08/24/14	--	1
Cobalt	6010B	<0.5	mg/kg		08/24/14	--	1
Copper	6010B	<2	mg/kg		08/24/14	--	1
Lead	6010B	<1	mg/kg		08/24/14	--	1
Mercury	7471A	<0.1	mg/kg			--	1
Molybdenum	6010B	<1	mg/kg		08/24/14	--	1
Nickel	6010B	<0.5	mg/kg		08/24/14	--	1
Selenium	6010B	<5	mg/kg		08/24/14	--	1
Silver	6010B	<0.5	mg/kg		08/24/14	--	1
Thallium	6010B	<5	mg/kg		08/24/14	--	1
Vanadium	6010B	<0.5	mg/kg		08/24/14	--	1
Zinc	6010B	<2	mg/kg		08/24/14	--	1

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Lab Reference #: OCA 15000
Project Name: OU3
Project #:

Organochlorinated Pesticides (EPA 8081A)

Client Sample ID	Lab Sample Number	Date Received	Date Sampled	Date Extracted	Date Analyzed	Matrix
Example Sample	15000-001	8/12/2014	8/12/2014		8/22/2014	Soil

ANALYTE	CAS #	µg/kg
Aldrin	309-00-2	<2
alpha-BHC	319-84-6	<5
beta-BHC	319-85-7	<5
delta-BHC	319-86-8	<10
gamma-BHC (Lindane)	58-89-9	<5
Chlordane	57-74-9	<30
4,4'-DDD	72-54-8	<10
4,4'-DDE	72-55-9	<5
4,4'-DDT	50-29-3	<10
Dieldrin	60-57-1	<2
Endosulfan I	959-98-8	<10
Endosulfan II	33213-65-9	<5
Endosulfan sulfate	1031-07-8	<10
Endrin	72-20-8	<10
Endrin aldehyde	7421-93-4	<10
Endrin ketone	53494-70-5	<5
Heptachlor	76-44-8	<2
Heptachlor epoxide	1024-57-3	<5
Methoxychlor	72-43-5	<10
Toxaphene	8001-35-2	<40

Surrogate:	% RC*
Decachlorobiphenyl	

* Acceptable Recovery: 35-152 %

Dilution Factor: 1

Data Qualifiers: None

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Lab Reference #: OCA 15000
Project Name: test
Project #: Tank CH7317

Polychlorinated Biphenyl's (EPA 8082)

Client Sample ID	Lab Sample Number	Date Received	Date Sampled	Date Extracted	Date Analyzed	Matrix
Tank CH7317	15000-001	8/12/2014 9:00	12/16/2014 12:15	2/12/2015 10:00	2/12/2015 17:56	Soil

ANALYTE	CAS #	µg/kg
PCB-1016	12674-11-2	<25
PCB-1221	11104-28-2	<25
PCB-1232	11141-16-5	<25
PCB-1242	53469-21-9	<25
PCB-1248	12672-29-6	<25
PCB-1254	11097-69-1	<25
PCB-1260	11096-82-5	<25

Surrogate:	% RC*
Decachlorobiphenyl	87

* Acceptable Recovery: 38-159 %

Dilution Factor: 1

Data Qualifiers: None

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Lab Reference #: OCA 15000
Project Name: OU3
Project #:

Volatile Organics by GC/MS (EPA 8260B)

Client Sample ID	Lab Sample Number	Date Received	Date Sampled	Date Extracted	Date Analyzed	Matrix
Example Sample	15000-001	8/12/2014	8/12/2014		8/22/2014	Soil

ANALYTE	CAS #	µg/kg	ANALYTE	CAS #	µg/kg
t-Amyl methyl ether (TAME)	994-05-8	<10	trans-1,3-Dichloropropene	10061-02-6	<2.5
Benzene	71-43-2	<2.0	Diisopropyl ether (DIPE)	108-20-3	<10
Bromobenzene	108-86-1	<2.5	Ethyl t-butyl ether (ETBE)	637-92-3	<10
Bromochloromethane	74-97-5	<2.5	Ethylbenzene	100-41-4	<2.5
Bromodichloromethane	75-27-4	<2.5	Hexachlorobutadiene	87-68-3	<5.0
Bromoform	75-25-2	<2.5	Isopropylbenzene	98-82-8	<2.5
Bromomethane	74-83-9	<10	4-Isopropyltoluene	99-87-6	<2.5
tert-Butyl alcohol (TBA)	75-65-0	<50	Methyl t-butyl ether (MTBE)	1634-04-4	<5.0
n-Butylbenzene	104-51-8	<2.5	Methylene chloride	75-09-2	<10
sec-Butylbenzene	135-98-8	<2.5	Naphthalene	91-20-3	<2.5
tert-Butylbenzene	98-06-6	<2.5	n-Propylbenzene	103-65-1	<2.5
Carbon tetrachloride	56-23-5	<2.5	Styrene	100-42-5	<2.5
Chlorobenzene	108-90-7	<2.5	1,1,1,2-Tetrachloroethane	630-20-6	<2.5
Chloroethane	75-00-3	<5.0	1,1,2,2-Tetrachloroethane	79-34-5	<2.5
Chloroform	67-66-3	<2.5	Tetrachloroethene	127-18-4	<2.5
Chloromethane	74-87-3	<5.0	Toluene	108-88-3	<2.5
2-Chlorotoluene	95-49-8	<2.5	1,2,3-Trichlorobenzene	87-61-6	<2.5
4-Chlorotoluene	106-43-4	<2.5	1,2,4-Trichlorobenzene	120-82-1	<2.5
Dibromochloromethane	124-48-1	<2.5	1,1,1-Trichloroethane	71-55-6	<2.5
1,2-Dibromo-3-chloropropane	96-12-8	<5.0	1,1,2-Trichloroethane	79-00-5	<2.5
1,2-Dibromoethane	106-93-4	<2.5	Trichloroethene	79-01-6	<2.5
Dibromomethane	74-95-3	<2.5	Trichlorofluoromethane	75-69-4	<5.0
1,2-Dichlorobenzene	95-50-1	<2.5	1,2,3-Trichloropropane	96-18-4	<2.5
1,3-Dichlorobenzene	541-73-1	<2.5	1,2,4-Trimethylbenzene	95-63-6	<2.5
1,4-Dichlorobenzene	106-46-7	<2.5	1,3,5-Trimethylbenzene	108-67-8	<2.5
Dichlorodifluoromethane	75-71-8	<2.5	Vinyl Chloride	75-01-4	<2.5
1,1-Dichloroethane	75-34-3	<2.5	Xylenes, Total	1330-20-7	<2.0
1,2-Dichloroethane	107-06-2	<2.5			
1,1-Dichloroethene	75-35-4	<2.5			
cis-1,2-Dichloroethene	156-59-2	<2.5			
trans-1,2-Dichloroethene	156-60-5	<2.5			
1,2-Dichloropropane	78-87-5	<2.5			
1,3-Dichloropropane	142-28-9	<2.5			
2,2-Dichloropropane	594-20-7	<2.5			
1,1-Dichloropropene	563-58-6	<2.5			
cis-1,3-Dichloropropene	10061-01-5	<2.5			

Surrogate:	% RC	Acceptable % RC
Dibromofluoromethane:		49-133 %
Toluene-d8:		62-130 %
4-Bromofluorobenzene:		55-130 %

Dilution Factor: 1

Data Qualifiers: None

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Lab Reference #: OCA 15000
Project Name: test
Project #:

Semi Volatile Organics by GC/MS (EPA 8270C)

Client Sample ID	Lab Sample Number	Date Received	Date Sampled	Date Extracted	Date Analyzed	Matrix
MW-1	15000-001	10/30/2015	10/29/2015	11/2/2015	11/3/2015	Soil

ANALYTE	CAS #	µg/kg	ANALYTE	CAS #	µg/kg
Acenaphthene:	83-32-9	<100	4,6-Dinitro-2-methylphenol:	534-52-1	<1000
Acenaphthylene:	208-96-8	<100	2,4-Dinitrophenol:	51-28-5	<1000
Aniline:	62-53-3	<100	2,4-Dinitrotoluene:	121-14-2	<250
Anthracene:	120-12-7	<100	2,6-Dinitrotoluene:	606-20-2	<250
Benz(a)anthracene:	56-55-3	<100	Di-n-octyl phthalate:	117-84-0	<100
Benzo(b)fluoranthene:	205-99-2	<100	Fluoranthene:	206-44-0	<100
Benzo(k)fluoranthene:	207-08-9	<100	Fluorene:	86-73-7	<100
Benzo(g,h,i)perylene:	191-24-2	<100	Hexachlorobenzene:	118-74-1	<100
Benzo(a)pyrene:	50-32-8	<100	Hexachlorobutadiene:	87-68-3	<100
Benzyl alcohol:	100-51-6	<100	Hexachlorocyclopentadiene:	77-47-4	<500
bis-(2-chloroethoxy) methane:	111-91-1	<100	Hexachloroethane:	67-72-1	<100
bis-(2-chloroethyl) ether:	111-44-4	<100	Indeno(1,2,3-cd)pyrene:	193-39-5	<100
bis-(2-chloroisopropyl) ether:	39638-32-9	<100	Isophorone:	78-59-1	<100
bis-(2-ethylhexyl) phthalate:	117-81-7	<100	2-Methylnaphthalene:	91-57-6	<100
4-Bromophenyl phenyl ether:	101-55-3	<100	2-Methylphenol:	95-48-7	<100
Butyl benzyl phthalate:	85-68-7	<100	3 & 4-Methylphenol:	108-39-4, 106-44-5	<100
4-Chloroaniline:	106-47-8	<100	Naphthalene:	91-20-3	<100
2-Chloronaphthalene:	91-58-7	<100	2-Nitroaniline:	88-74-4	<250
4-Chloro-3-methylphenol:	59-50-7	<100	3-Nitroaniline:	99-09-2	<250
2-Chlorophenol:	95-57-8	<100	4-Nitroaniline:	100-01-6	<250
4-Chlorophenyl phenyl ether:	7005-72-3	<100	Nitrobenzene:	98-95-3	<100
Chrysene:	218-01-9	<100	2-Nitrophenol:	88-75-5	<100
Dibenz(a,h)anthracene:	53-70-3	<100	4-Nitrophenol:	100-02-7	<1000
Dibenzofuran:	132-64-9	<100	N-Nitrosodiphenylamine:	86-30-6	<100
Di-n-butyl phthalate:	84-74-2	<100	N-Nitrosodi-n-propylamine:	621-64-7	<100
1,2-Dichlorobenzene:	95-50-1	<100	N-Nitrosodimethylamine:	62-75-9	<100
1,3-Dichlorobenzene:	541-73-1	<100	Pentachlorophenol:	87-86-5	<500
1,4-Dichlorobenzene:	106-46-7	<100	Phenanthrene:	85-01-8	<100
2,4-Dichlorophenol:	120-83-2	<100	Phenol:	108-95-2	<100
Diethyl phthalate:	84-66-2	<100	Pyrene:	129-00-0	<100
2,4-Dimethylphenol:	105-67-9	<100	1,2,4-Trichlorobenzene:	120-82-1	<100
Dimethyl phthalate:	131-11-3	<100	2,4,5-Trichlorophenol:	95-95-4	<100
			2,4,6-Trichlorophenol:	88-06-2	<100

Surrogate:	% RC	Acceptable % RC
2-Fluorophenol:	56	25-130 %
Phenol-d6:	67	35-130 %
Nitrobenzene-d5:	85	36-130 %
2-Fluorobiphenyl:	77	39-130 %
2,4,6-Tribromophenol:	85	32-135 %
Terphenyl-d14:	79	48-140 %

Dilution Factor: 1

Data Qualifiers: None

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Lab Reference #: OCA 15000
Project Name: test
Project #: Tank CH7317

Polynuclear Aromatic Hydrocarbons (EPA 8310)

Client Sample ID	Lab Sample Number	Date Received	Date Sampled	Date Extracted	Date Analyzed	Matrix
Tank CH7317	15000-001	8/12/2014 9:00	12/16/2014 12:15	10/23/2013 8:00	10/23/2013 15:00	Soil

ANALYTE	CAS #	µg/kg
Acenaphthene:	83-32-9	<2
Acenaphthylene:	208-96-8	<5
Anthracene:	120-12-7	<2
Benz(a)anthracene:	56-55-3	<2
Benzo(a)pyrene:	50-32-8	<2
Benzo(b)fluoranthene:	205-99-2	<2
Benzo(k)fluoranthene:	207-08-9	<2
Benzo(g,h,i)perylene:	191-24-2	<2
Chrysene:	218-01-9	<2
Dibenz(a,h)anthracene:	53-70-3	<2
Fluoranthene:	206-44-0	<2
Pyrene:	129-00-0	<2
Fluorene:	86-73-7	<10
Phenanthrene:	85-01-8	<2
Indeno(1,2,3-cd)pyrene:	193-39-5	<2
Naphthalene:	91-20-3	<5

Surrogate:	% RC*
Nitrobenzene-d5	100

* Acceptable Recovery: 40-130 %

Dilution Factor: 1

Data Qualifiers: None

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Lab Reference #: OCA 15000
Project Name: test
Project #:

Gasoline Range Organics - GROs (EPA M8015B)

Client Sample ID	Lab Sample Number	Date Received	Date Sampled	Date Extracted	Date Analyzed	Matrix
MW-1	15000-001	10/30/2015 9:00	10/29/2015 12:15	11/17/2015 15:00	11/17/2015 19:00	Soil

ANALYTE	mg/kg
TPH as GROs(C4-C12)	<0.25

Surrogate:	% RC*
α-α-α-Trifluorotoluene	

Dilution Factor: 1
Data Qualifiers: None

* Acceptable Recovery: 51-130 %

Gasoline Range Organics (GROs) are quantitated against a gasoline standard.

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Lab Reference #: OCA 15000
Project Name: test
Project #: Tank CH7317

C10-C32 Diesel (CADHS LUFT)

Client Sample ID	Lab Sample Number	Date Received	Date Sampled	Date Extracted	Date Analyzed	Matrix
water test	15000-002	3/25/2012		2/12/2015	2/12/2015	Water

ANALYTE	mg/L
C12-C22	<0.5

Surrogate: % RC*
Octacosane

* Acceptable Recovery: 46-161 %

Dilution Factor: 1
Data Qualifiers: None

APPENDIX B

EPA METHOD 6200

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the in situ and intrusive analysis of the 26 analytes listed below for soil and sediment samples. Some common elements are not listed in this method because they are considered "light" elements that cannot be detected by field portable x-ray fluorescence (FPXRF). These light elements are: lithium, beryllium, sodium, magnesium, aluminum, silicon, and phosphorus. Most of the analytes listed below are of environmental concern, while a few others have interference effects or change the elemental composition of the matrix, affecting quantitation of the analytes of interest. Generally elements of atomic number 16 or greater can be detected and quantitated by FPXRF. The following RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Antimony (Sb)	7440-36-0
Arsenic (As)	7440-38-0
Barium (Ba)	7440-39-3
Cadmium (Cd)	7440-43-9
Chromium (Cr)	7440-47-3
Cobalt (Co)	7440-48-4
Copper (Cu)	7440-50-8
Lead (Pb)	7439-92-1
Mercury (Hg)	7439-97-6
Nickel (Ni)	7440-02-0
Selenium (Se)	7782-49-2
Silver (Ag)	7440-22-4
Thallium (Tl)	7440-28-0
Tin (Sn)	7440-31-5

Analytes	CAS Registry No.
Vanadium (V)	7440-62-2
Zinc (Zn)	7440-66-6

In addition, the following non-RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Calcium (Ca)	7440-70-2
Iron (Fe)	7439-89-6
Manganese (Mn)	7439-96-5
Molybdenum (Mo)	7439-93-7
Potassium (K)	7440-09-7
Rubidium (Rb)	7440-17-7
Strontium (Sr)	7440-24-6
Thorium (Th)	7440-29-1
Titanium (Ti)	7440-32-6
Zirconium (Zr)	7440-67-7

1.2 This method is a screening method to be used with confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnance atomic absorption spectrometry (GFAA), inductively coupled plasma-atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)). This method's main strength is that it is a rapid field screening procedure. The method's lower limits of detection are typically above the toxicity characteristic regulatory level for most RCRA analytes. However, when the obtainable values for precision, accuracy, and laboratory-established sensitivity of this method meet project-specific data quality objectives (DQOs), FPXRF is a fast, powerful, cost effective technology for site characterization.

1.3 The method sensitivity or lower limit of detection depends on several factors, including the analyte of interest, the type of detector used, the type of excitation source, the strength of the excitation source, count times used to irradiate the sample, physical matrix effects, chemical matrix effects, and interelement spectral interferences. Example lower limits of detection for analytes of interest in environmental applications are shown in Table 1. These limits apply to a clean spiked matrix of quartz sand (silicon dioxide) free of interelement spectral interferences using long (100 -600 second) count times. These sensitivity values are given for guidance only and may not always be achievable, since they will vary depending on the sample matrix, which instrument is used, and operating conditions. A discussion of performance-based sensitivity is presented in Sec. 9.6.

1.4 Analysts should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.5 Use of this method is restricted to use by, or under supervision of, personnel appropriately experienced and trained in the use and operation of an XRF instrument. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The FPXRF technologies described in this method use either sealed radioisotope sources or x-ray tubes to irradiate samples with x-rays. When a sample is irradiated with x-rays, the source x-rays may undergo either scattering or absorption by sample atoms. This latter process is known as the photoelectric effect. When an atom absorbs the source x-rays, the incident radiation dislodges electrons from the innermost shells of the atom, creating vacancies. The electron vacancies are filled by electrons cascading in from outer electron shells. Electrons in outer shells have higher energy states than inner shell electrons, and the outer shell electrons give off energy as they cascade down into the inner shell vacancies. This rearrangement of electrons results in emission of x-rays characteristic of the given atom. The emission of x-rays, in this manner, is termed x-ray fluorescence.

Three electron shells are generally involved in emission of x-rays during FPXRF analysis of environmental samples. The three electron shells include the K, L, and M shells. A typical emission pattern, also called an emission spectrum, for a given metal has multiple intensity peaks generated from the emission of K, L, or M shell electrons. The most commonly measured x-ray emissions are from the K and L shells; only metals with an atomic number greater than 57 have measurable M shell emissions.

Each characteristic x-ray line is defined with the letter K, L, or M, which signifies which shell had the original vacancy and by a subscript alpha (α), beta (β), or gamma (γ) etc., which indicates the higher shell from which electrons fell to fill the vacancy and produce the x-ray. For example, a K_α line is produced by a vacancy in the K shell filled by an L shell electron, whereas a K_β line is produced by a vacancy in the K shell filled by an M shell electron. The K_α transition is on average 6 to 7 times more probable than the K_β transition; therefore, the K_α line is approximately 7 times more intense than the K_β line for a given element, making the K_α line the choice for quantitation purposes.

The K lines for a given element are the most energetic lines and are the preferred lines for analysis. For a given atom, the x-rays emitted from L transitions are always less energetic than those emitted from K transitions. Unlike the K lines, the main L emission lines (L_α and L_β) for an element are of nearly equal intensity. The choice of one or the other depends on what interfering element lines might be present. The L emission lines are useful for analyses involving elements of atomic number (Z) 58 (cerium) through 92 (uranium).

An x-ray source can excite characteristic x-rays from an element only if the source energy is greater than the absorption edge energy for the particular line group of the element, that is, the K absorption edge, L absorption edge, or M absorption edge energy. The absorption edge energy is somewhat greater than the corresponding line energy. Actually, the K absorption edge energy is approximately the sum of the K, L, and M line energies of the particular element, and the L absorption edge energy is approximately the sum of the L and M line energies. FPXRF is more sensitive to an element with an absorption edge energy close to but less than

the excitation energy of the source. For example, when using a cadmium-109 source, which has an excitation energy of 22.1 kiloelectron volts (keV), FPXRF would exhibit better sensitivity for zirconium which has a K line energy of 15.77 keV than to chromium, which has a K line energy of 5.41 keV.

2.2 Under this method, inorganic analytes of interest are identified and quantitated using a field portable energy-dispersive x-ray fluorescence spectrometer. Radiation from one or more radioisotope sources or an electrically excited x-ray tube is used to generate characteristic x-ray emissions from elements in a sample. Up to three sources may be used to irradiate a sample. Each source emits a specific set of primary x-rays that excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the source is selected according to its excitation efficiency for the element of interest.

For measurement, the sample is positioned in front of the probe window. This can be done in two manners using FPXRF instruments, specifically, in situ or intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. The sample cup is then placed on top of the window inside a protective cover for analysis.

Sample analysis is then initiated by exposing the sample to primary radiation from the source. Fluorescent and backscattered x-rays from the sample enter through the detector window and are converted into electric pulses in the detector. The detector in FPXRF instruments is usually either a solid-state detector or a gas-filled proportional counter. Within the detector, energies of the characteristic x-rays are converted into a train of electric pulses, the amplitudes of which are linearly proportional to the energy of the x-rays. An electronic multichannel analyzer (MCA) measures the pulse amplitudes, which is the basis of qualitative x-ray analysis. The number of counts at a given energy per unit of time is representative of the element concentration in a sample and is the basis for quantitative analysis. Most FPXRF instruments are menu-driven from software built into the units or from personal computers (PC).

The measurement time of each source is user-selectable. Shorter source measurement times (30 seconds) are generally used for initial screening and hot spot delineation, and longer measurement times (up to 300 seconds) are typically used to meet higher precision and accuracy requirements.

FPXRF instruments can be calibrated using the following methods: internally using fundamental parameters determined by the manufacturer, empirically based on site-specific calibration standards (SSCS), or based on Compton peak ratios. The Compton peak is produced by backscattering of the source radiation. Some FPXRF instruments can be calibrated using multiple methods.

3.0 DEFINITIONS

3.1 FPXRF -- Field portable x-ray fluorescence.

3.2 MCA -- Multichannel analyzer for measuring pulse amplitude.

3.3 SSCS -- Site-specific calibration standards.

3.4 FP -- Fundamental parameter.

3.5 ROI -- Region of interest.

3.6 SRM -- Standard reference material; a standard containing certified amounts of metals in soil or sediment.

3.7 eV -- Electron volt; a unit of energy equivalent to the amount of energy gained by an electron passing through a potential difference of one volt.

3.8 Refer to Chapter One, Chapter Three, and the manufacturer's instructions for other definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 The total method error for FPXRF analysis is defined as the square root of the sum of squares of both instrument precision and user- or application-related error. Generally, instrument precision is the least significant source of error in FPXRF analysis. User- or application-related error is generally more significant and varies with each site and method used. Some sources of interference can be minimized or controlled by the instrument operator, but others cannot. Common sources of user- or application-related error are discussed below.

4.2 Physical matrix effects result from variations in the physical character of the sample. These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, if any analyte exists in the form of very fine particles in a coarser-grained matrix, the analyte's concentration measured by the FPXRF will vary depending on how fine particles are distributed within the coarser-grained matrix. If the fine particles "settle" to the bottom of the sample cup (i.e., against the cup window), the analyte concentration measurement will be higher than if the fine particles are not mixed in well and stay on top of the coarser-grained particles in the sample cup. One way to reduce such error is to grind and sieve all soil samples to a uniform particle size thus reducing sample-to-sample particle size variability. Homogeneity is always a concern when dealing with soil samples. Every effort should be made to thoroughly mix and homogenize soil samples before analysis. Field studies have shown heterogeneity of the sample generally has the largest impact on comparability with confirmatory samples.

4.3 Moisture content may affect the accuracy of analysis of soil and sediment sample analyses. When the moisture content is between 5 and 20 percent, the overall error from moisture may be minimal. However, moisture content may be a major source of error when analyzing samples of surface soil or sediment that are saturated with water. This error can be minimized by drying the samples in a convection or toaster oven. Microwave drying is not recommended because field studies have shown that microwave drying can increase variability between FPXRF data and confirmatory analysis and because metal fragments in the sample can cause arcing to occur in a microwave.

4.4 Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases as the distance from the radioactive source increases. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample should be flat and smooth to provide a good contact surface.

4.5 Chemical matrix effects result from differences in the concentrations of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as x-ray absorption and enhancement phenomena. Both effects are common in soils contaminated with heavy metals. As examples of absorption and enhancement effects; iron (Fe) tends to absorb copper (Cu) x-rays, reducing the intensity of the Cu measured by the detector, while chromium (Cr) will be enhanced at the expense of Fe because the absorption edge of Cr is slightly lower in energy than the fluorescent peak of iron. The effects can be corrected mathematically through the use of fundamental parameter (FP) coefficients. The effects also can be compensated for using SSCS, which contain all the elements present on site that can interfere with one another.

4.6 When present in a sample, certain x-ray lines from different elements can be very close in energy and, therefore, can cause interference by producing a severely overlapped spectrum. The degree to which a detector can resolve the two different peaks depends on the energy resolution of the detector. If the energy difference between the two peaks in electron volts is less than the resolution of the detector in electron volts, then the detector will not be able to fully resolve the peaks.

The most common spectrum overlaps involve the K_β line of element Z-1 with the K_α line of element Z. This is called the K_α/K_β interference. Because the K_α:K_β intensity ratio for a given element usually is about 7:1, the interfering element, Z-1, must be present at large concentrations to cause a problem. Two examples of this type of spectral interference involve the presence of large concentrations of vanadium (V) when attempting to measure Cr or the presence of large concentrations of Fe when attempting to measure cobalt (Co). The V K_α and K_β energies are 4.95 and 5.43 keV, respectively, and the Cr K_α energy is 5.41 keV. The Fe K_α and K_β energies are 6.40 and 7.06 keV, respectively, and the Co K_α energy is 6.92 keV. The difference between the V K_β and Cr K_α energies is 20 eV, and the difference between the Fe K_β and the Co K_α energies is 140 eV. The resolution of the highest-resolution detectors in FPXRF instruments is 170 eV. Therefore, large amounts of V and Fe will interfere with quantitation of Cr or Co, respectively. The presence of Fe is a frequent problem because it is often found in soils at tens of thousands of parts per million (ppm).

4.7 Other interferences can arise from K/L, K/M, and L/M line overlaps, although these overlaps are less common. Examples of such overlap involve arsenic (As) K_α/lead (Pb) L_α and sulfur (S) K_α/Pb M_α. In the As/Pb case, Pb can be measured from the Pb L_β line, and As can be measured from either the As K_α or the As $K_ß$ line; in this way the interference can be corrected. If the As K_β line is used, sensitivity will be decreased by a factor of two to five times because it is a less intense line than the As K_α line. If the As K_α line is used in the presence of Pb, mathematical corrections within the instrument software can be used to subtract out the Pb interference. However, because of the limits of mathematical corrections, As concentrations cannot be efficiently calculated for samples with Pb:As ratios of 10:1 or more. This high ratio of Pb to As may result in reporting of a "nondetect" or a "less than" value (e.g., <300 ppm) for As, regardless of the actual concentration present.

No instrument can fully compensate for this interference. It is important for an operator to understand this limitation of FPXRF instruments and consult with the manufacturer of the FPXRF instrument to evaluate options to minimize this limitation. The operator's decision will be based on action levels for metals in soil established for the site, matrix effects, capabilities of the instrument, data quality objectives, and the ratio of lead to arsenic known to be present at the site. If a site is encountered that contains lead at concentrations greater than ten times the concentration of arsenic it is advisable that all critical soil samples be sent off site for confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnance atomic absorption spectrometry (GFAA), inductively coupled plasma-

atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)).

4.8 If SSCS are used to calibrate an FPXRF instrument, the samples collected must be representative of the site under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentrations of the contaminants of concern at a given time and location. Analytical results for representative samples reflect variations in the presence and concentration ranges of contaminants throughout a site. Variables affecting sample representativeness include differences in soil type, contaminant concentration variability, sample collection and preparation variability, and analytical variability, all of which should be minimized as much as possible.

4.9 Soil physical and chemical effects may be corrected using SSCS that have been analyzed by inductively coupled plasma (ICP) or atomic absorption (AA) methods. However, a major source of error can be introduced if these samples are not representative of the site or if the analytical error is large. Another concern is the type of digestion procedure used to prepare the soil samples for the reference analysis. Analytical results for the confirmatory method will vary depending on whether a partial digestion procedure, such as Method 3050, or a total digestion procedure, such as Method 3052, is used. It is known that depending on the nature of the soil or sediment, Method 3050 will achieve differing extraction efficiencies for different analytes of interest. The confirmatory method should meet the project-specific data quality objectives (DQOs).

XRF measures the total concentration of an element; therefore, to achieve the greatest comparability of this method with the reference method (reduced bias), a total digestion procedure should be used for sample preparation. However, in the study used to generate the performance data for this method (see Table 8), the confirmatory method used was Method 3050, and the FPXRF data compared very well with regression correlation coefficients (r often exceeding 0.95, except for barium and chromium). The critical factor is that the digestion procedure and analytical reference method used should meet the DQOs of the project and match the method used for confirmation analysis.

4.10 Ambient temperature changes can affect the gain of the amplifiers producing instrument drift. Gain or drift is primarily a function of the electronics (amplifier or preamplifier) and not the detector as most instrument detectors are cooled to a constant temperature. Most FPXRF instruments have a built-in automatic gain control. If the automatic gain control is allowed to make periodic adjustments, the instrument will compensate for the influence of temperature changes on its energy scale. If the FPXRF instrument has an automatic gain control function, the operator will not have to adjust the instrument's gain unless an error message appears. If an error message appears, the operator should follow the manufacturer's procedures for troubleshooting the problem. Often, this involves performing a new energy calibration. The performance of an energy calibration check to assess drift is a quality control measure discussed in Sec. 9.2.

If the operator is instructed by the manufacturer to manually conduct a gain check because of increasing or decreasing ambient temperature, it is standard to perform a gain check after every 10 to 20 sample measurements or once an hour whichever is more frequent. It is also suggested that a gain check be performed if the temperature fluctuates more than 10E F. The operator should follow the manufacturer's recommendations for gain check frequency.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The user is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

NOTE: No MSDS applies directly to the radiation-producing instrument because that is covered under the Nuclear Regulatory Commission (NRC) or applicable state regulations.

5.2 Proper training for the safe operation of the instrument and radiation training should be completed by the analyst prior to analysis. Radiation safety for each specific instrument can be found in the operator's manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer. The analyst should be aware of the local state and national regulations that pertain to the use of radiation-producing equipment and radioactive materials with which compliance is required. There should be a person appointed within the organization that is solely responsible for properly instructing all personnel, maintaining inspection records, and monitoring x-ray equipment at regular intervals.

Licenses for radioactive materials are of two types, specifically: (1) a general license which is usually initiated by the manufacturer for receiving, acquiring, owning, possessing, using, and transferring radioactive material incorporated in a device or equipment, and (2) a specific license which is issued to named persons for the operation of radioactive instruments as required by local, state, or federal agencies. A copy of the radioactive material license (for specific licenses only) and leak tests should be present with the instrument at all times and available to local and national authorities upon request.

X-ray tubes do not require radioactive material licenses or leak tests, but do require approvals and licenses which vary from state to state. In addition, fail-safe x-ray warning lights should be illuminated whenever an x-ray tube is energized. Provisions listed above concerning radiation safety regulations, shielding, training, and responsible personnel apply to x-ray tubes just as to radioactive sources. In addition, a log of the times and operating conditions should be kept whenever an x-ray tube is energized. An additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply, however, if the tube is properly positioned within the instrument, this is only a negligible risk. Any instrument (x-ray tube or radioisotope based) is capable of delivering an electric shock from the basic circuitry when the system is inappropriately opened.

5.3 Radiation monitoring equipment should be used with the handling and operation of the instrument. The operator and the surrounding environment should be monitored continually for analyst exposure to radiation. Thermal luminescent detectors (TLD) in the form of badges and rings are used to monitor operator radiation exposure. The TLDs or badges should be worn in the area of maximum exposure. The maximum permissible whole-body dose from occupational exposure is 5 Roentgen Equivalent Man (REM) per year. Possible exposure pathways for radiation to enter the body are ingestion, inhaling, and absorption. The best precaution to prevent radiation exposure is distance and shielding.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for

use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

6.1 FPXRF spectrometer -- An FPXRF spectrometer consists of four major components: (1) a source that provides x-rays; (2) a sample presentation device; (3) a detector that converts x-ray-generated photons emitted from the sample into measurable electronic signals; and (4) a data processing unit that contains an emission or fluorescence energy analyzer, such as an MCA, that processes the signals into an x-ray energy spectrum from which elemental concentrations in the sample may be calculated, and a data display and storage system. These components and additional, optional items, are discussed below.

6.1.1 Excitation sources -- FPXRF instruments use either a sealed radioisotope source or an x-ray tube to provide the excitation source. Many FPXRF instruments use sealed radioisotope sources to produce x-rays in order to irradiate samples. The FPXRF instrument may contain between one and three radioisotope sources. Common radioisotope sources used for analysis for metals in soils are iron Fe-55 (^{55}Fe), cadmium Cd-109 (^{109}Cd), americium Am-241 (^{241}Am), and curium Cm-244 (^{244}Cm). These sources may be contained in a probe along with a window and the detector; the probe may be connected to a data reduction and handling system by means of a flexible cable. Alternatively, the sources, window, and detector may be included in the same unit as the data reduction and handling system.

The relative strength of the radioisotope sources is measured in units of millicuries (mCi). All other components of the FPXRF system being equal, the stronger the source, the greater the sensitivity and precision of a given instrument. Radioisotope sources undergo constant decay. In fact, it is this decay process that emits the primary x-rays used to excite samples for FPXRF analysis. The decay of radioisotopes is measured in "half-lives." The half-life of a radioisotope is defined as the length of time required to reduce the radioisotopes strength or activity by half. Developers of FPXRF technologies recommend source replacement at regular intervals based on the source's half-life. This is due to the ever increasing time required for the analysis rather than a decrease in instrument performance. The characteristic x-rays emitted from each of the different sources have energies capable of exciting a certain range of analytes in a sample. Table 2 summarizes the characteristics of four common radioisotope sources.

X-ray tubes have higher radiation output, no intrinsic lifetime limit, produce constant output over their lifetime, and do not have the disposal problems of radioactive sources but are just now appearing in FPXRF instruments. An electrically-excited x-ray tube operates by bombarding an anode with electrons accelerated by a high voltage. The electrons gain an energy in electron volts equal to the accelerating voltage and can excite atomic transitions in the anode, which then produces characteristic x-rays. These characteristic x-rays are emitted through a window which contains the vacuum necessary for the electron acceleration. An important difference between x-ray tubes and radioactive sources is that the electrons which bombard the anode also produce a continuum of x-rays across a broad range of energies in addition to the characteristic x-rays. This continuum is weak compared to the characteristic x-rays but can provide substantial excitation since it covers a broad energy range. It has the undesired property of producing background in the spectrum near the analyte x-ray lines when it is scattered by the sample. For this reason a filter is often used between the x-ray tube and the sample to suppress the continuum radiation while passing the characteristic x-rays from the anode. This filter is sometimes incorporated into the window of the x-ray tube. The choice of

accelerating voltage is governed both by the anode material, since the electrons must have sufficient energy to excite the anode, which requires a voltage greater than the absorption edge of the anode material and by the instrument's ability to cool the x-ray tube. The anode is most efficiently excited by voltages 2 to 2.5 times the edge energy (most x-rays per unit power to the tube), although voltages as low as 1.5 times the absorption edge energy will work. The characteristic x-rays emitted by the anode are capable of exciting a range of elements in the sample just as with a radioactive source. Table 3 gives the recommended operating voltages and the sample elements excited for some common anodes.

6.1.2 Sample presentation device -- FPXRF instruments can be operated in two modes: in situ and intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. For FPXRF instruments operated in the intrusive mode, the probe may be rotated so that the window faces either upward or downward. A protective sample cover is placed over the window, and the sample cup is placed on top of the window inside the protective sample cover for analysis.

6.1.3 Detectors -- The detectors in the FPXRF instruments can be either solid-state detectors or gas-filled, proportional counter detectors. Common solid-state detectors include mercuric iodide (HgI_2), silicon pin diode and lithium-drifted silicon Si(Li). The HgI_2 detector is operated at a moderately subambient temperature controlled by a low power thermoelectric cooler. The silicon pin diode detector also is cooled via the thermoelectric Peltier effect. The Si(Li) detector must be cooled to at least -90 EC either with liquid nitrogen or by thermoelectric cooling via the Peltier effect. Instruments with a Si(Li) detector have an internal liquid nitrogen dewar with a capacity of 0.5 to 1.0 L. Proportional counter detectors are rugged and lightweight, which are important features of a field portable detector. However, the resolution of a proportional counter detector is not as good as that of a solid-state detector. The energy resolution of a detector for characteristic x-rays is usually expressed in terms of full width at half-maximum (FWHM) height of the manganese K_α peak at 5.89 keV. The typical resolutions of the above mentioned detectors are as follows: HgI_2-270 eV; silicon pin diode-250 eV; Si(Li)–170 eV; and gas-filled, proportional counter-750 eV.

During operation of a solid-state detector, an x-ray photon strikes a biased, solid-state crystal and loses energy in the crystal by producing electron-hole pairs. The electric charge produced is collected and provides a current pulse that is directly proportional to the energy of the x-ray photon absorbed by the crystal of the detector. A gas-filled, proportional counter detector is an ionization chamber filled with a mixture of noble and other gases. An x-ray photon entering the chamber ionizes the gas atoms. The electric charge produced is collected and provides an electric signal that is directly proportional to the energy of the x-ray photon absorbed by the gas in the detector.

6.1.4 Data processing units -- The key component in the data processing unit of an FPXRF instrument is the MCA. The MCA receives pulses from the detector and sorts them by their amplitudes (energy level). The MCA counts pulses per second to determine the height of the peak in a spectrum, which is indicative of the target analyte's concentration. The spectrum of element peaks are built on the MCA. The MCAs in FPXRF instruments have from 256 to 2,048 channels. The concentrations of target analytes are usually shown in ppm on a liquid crystal display (LCD) in the instrument. FPXRF instruments can store both spectra and from 3,000 to 5,000 sets of numerical analytical results. Most FPXRF instruments are menu-driven from software built into the

units or from PCs. Once the data–storage memory of an FPXRF unit is full or at any other time, data can be downloaded by means of an RS-232 port and cable to a PC.

6.2 Spare battery and battery charger.

6.3 Polyethylene sample cups -- 31 to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

6.4 X-ray window film -- Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 µm thick.

6.5 Mortar and pestle -- Glass, agate, or aluminum oxide; for grinding soil and sediment samples.

6.6 Containers -- Glass or plastic to store samples.

6.7 Sieves -- 60-mesh (0.25 mm), stainless-steel, Nylon, or equivalent for preparing soil and sediment samples.

6.8 Trowels -- For smoothing soil surfaces and collecting soil samples.

6.9 Plastic bags -- Used for collection and homogenization of soil samples.

6.10 Drying oven -- Standard convection or toaster oven, for soil and sediment samples that require drying.

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Pure element standards -- Each pure, single-element standard is intended to produce strong characteristic x-ray peaks of the element of interest only. Other elements present must not contribute to the fluorescence spectrum. A set of pure element standards for commonly sought analytes is supplied by the instrument manufacturer, if designated for the instrument; not all instruments require the pure element standards. The standards are used to set the region of interest (ROI) for each element. They also can be used as energy calibration and resolution check samples.

7.3 Site-specific calibration standards -- Instruments that employ fundamental parameters (FP) or similar mathematical models in minimizing matrix effects may not require SSCS. If the FP calibration model is to be optimized or if empirical calibration is necessary, then SSCSs must be collected, prepared, and analyzed.

7.3.1 The SSCS must be representative of the matrix to be analyzed by FPXRF. These samples must be well homogenized. A minimum of 10 samples spanning the concentration ranges of the analytes of interest and of the interfering elements must be obtained from the site. A sample size of 4 to 8 ounces is recommended, and standard glass sampling jars should be used.

7.3.2 Each sample should be oven-dried for 2 to 4 hr at a temperature of less than 150 EC. If mercury is to be analyzed, a separate sample portion should be dried at ambient temperature as heating may volatilize the mercury. When the sample is dry, all large, organic debris and nonrepresentative material, such as twigs, leaves, roots, insects, asphalt, and rock should be removed. The sample should be homogenized (see Sec. 7.3.3) and then a representative portion ground with a mortar and pestle or other mechanical means, prior to passing through a 60-mesh sieve. Only the coarse rock fraction should remain on the screen.

7.3.3 The sample should be homogenized by using a riffle splitter or by placing 150 to 200 g of the dried, sieved sample on a piece of kraft or butcher paper about 1.5 by 1.5 feet in size. Each corner of the paper should be lifted alternately, rolling the soil over on itself and toward the opposite corner. The soil should be rolled on itself 20 times. Approximately 5 g of the sample should then be removed and placed in a sample cup for FPXRF analysis. The rest of the prepared sample should be sent off site for ICP or AA analysis. The method use for confirmatory analysis should meet the data quality objectives of the project.

7.4 Blank samples -- The blank samples should be from a "clean" quartz or silicon dioxide matrix that is free of any analytes at concentrations above the established lower limit of detection. These samples are used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

7.5 Standard reference materials -- Standard reference materials (SRMs) are standards containing certified amounts of metals in soil or sediment. These standards are used for accuracy and performance checks of FPXRF analyses. SRMs can be obtained from the National Institute of Standards and Technology (NIST), the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories. When these SRMs are unavailable, alternate standards may be used (e.g., NIST 2702).

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Sample handling and preservation procedures used in FPXRF analyses should follow the guidelines in Chapter Three, "Inorganic Analytes."

9.0 QUALITY CONTROL

9.1 Follow the manufacturer's instructions for the quality control procedures specific to use of the testing product. Refer to Chapter One for additional guidance on quality assurance (QA) and quality control (QC) protocols. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results.

9.2 Energy calibration check -- To determine whether an FPXRF instrument is operating within resolution and stability tolerances, an energy calibration check should be run. The energy calibration check determines whether the characteristic x-ray lines are shifting,

which would indicate drift within the instrument. As discussed in Sec. 4.10, this check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (more than 10 EF).

9.2.1 The energy calibration check should be run at a frequency consistent with manufacturer's recommendations. Generally, this would be at the beginning of each working day, after the batteries are changed or the instrument is shut off, at the end of each working day, and at any other time when the instrument operator believes that drift is occurring during analysis. A pure element such as iron, manganese, copper, or lead is often used for the energy calibration check. A manufacturer-recommended count time per source should be used for the check.

9.2.2 The instrument manufacturer's manual specifies the channel or kiloelectron volt level at which a pure element peak should appear and the expected intensity of the peak. The intensity and channel number of the pure element as measured using the source should be checked and compared to the manufacturer's recommendation. If the energy calibration check does not meet the manufacturer's criteria, then the pure element sample should be repositioned and reanalyzed. If the criteria are still not met, then an energy calibration should be performed as described in the manufacturer's manual. With some FPXRF instruments, once a spectrum is acquired from the energy calibration check, the peak can be optimized and realigned to the manufacturer's specifications using their software.

9.3 Blank samples -- Two types of blank samples should be analyzed for FPXRF analysis, specifically, instrument blanks and method blanks.

9.3.1 An instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window. The instrument blank can be silicon dioxide, a polytetraflurorethylene (PTFE) block, a quartz block, "clean" sand, or lithium carbonate. This instrument blank should be analyzed on each working day before and after analyses are conducted and once per every twenty samples. An instrument blank should also be analyzed whenever contamination is suspected by the analyst. The frequency of analysis will vary with the data quality objectives of the project. A manufacturer-recommended count time per source should be used for the blank analysis. No element concentrations above the established lower limit of detection should be found in the instrument blank. If concentrations exceed these limits, then the probe window and the check sample should be checked for contamination. If contamination is not a problem, then the instrument must be "zeroed" by following the manufacturer's instructions.

9.3.2 A method blank is used to monitor for laboratory-induced contaminants or interferences. The method blank can be "clean" silica sand or lithium carbonate that undergoes the same preparation procedure as the samples. A method blank must be analyzed at least daily. The frequency of analysis will depend on the data quality objectives of the project. If the method blank does not contain the target analyte at a level that interferes with the project-specific data quality objectives then the method blank would be considered acceptable. In the absence of project-specific data quality objectives, if the blank is less than the lowest level of detection or less than 10% of the lowest sample concentration for the analyte, whichever is greater, then the method blank would be considered acceptable. If the method blank cannot be considered acceptable, the cause of the problem must be identified, and all samples analyzed with the method blank must be reanalyzed.

9.4 Calibration verification checks -- A calibration verification check sample is used to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest. A check sample should be analyzed at the beginning of each working day, during active sample analyses, and at the end of each working day. The frequency of calibration checks during active analysis will depend on the data quality objectives of the project. The check sample should be a well characterized soil sample from the site that is representative of site samples in terms of particle size and degree of homogeneity and that contains contaminants at concentrations near the action levels. If a site-specific sample is not available, then an NIST or other SRM that contains the analytes of interest can be used to verify the accuracy of the instrument. The measured value for each target analyte should be within ±20 percent (%D) of the true value for the calibration verification check to be acceptable. If a measured value falls outside this range, then the check sample should be reanalyzed. If the value continues to fall outside the acceptance range, the instrument should be recalibrated, and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

9.5 Precision measurements -- The precision of the method is monitored by analyzing a sample with low, moderate, or high concentrations of target analytes. The frequency of precision measurements will depend on the data quality objectives for the data. A minimum of one precision sample should be run per day. Each precision sample should be analyzed 7 times in replicate. It is recommended that precision measurements be obtained for samples with varying concentration ranges to assess the effect of concentration on method precision. Determining method precision for analytes at concentrations near the site action levels can be extremely important if the FPXRF results are to be used in an enforcement action; therefore, selection of at least one sample with target analyte concentrations at or near the site action levels or levels of concern is recommended. A precision sample is analyzed by the instrument for the same field analysis time as used for other project samples. The relative standard deviation (RSD) of the sample mean is used to assess method precision. For FPXRF data to be considered adequately precise, the RSD should not be greater than 20 percent with the exception of chromium. RSD values for chromium should not be greater than 30 percent. If both in situ and intrusive analytical techniques are used during the course of one day, it is recommended that separate precision calculations be performed for each analysis type.

The equation for calculating RSD is as follows:

$$RSD = (SD/Mean\ Concentration) \times 100$$

where:

RSD = Relative standard deviation for the precision measurement for the analyte
SD = Standard deviation of the concentration for the analyte
Mean concentration = Mean concentration for the analyte

The precision or reproducibility of a measurement will improve with increasing count time, however, increasing the count time by a factor of 4 will provide only 2 times better precision, so there is a point of diminishing return. Increasing the count time also improves the sensitivity, but decreases sample throughput.

9.6 The lower limits of detection should be established from actual measured performance based on spike recoveries in the matrix of concern or from acceptable method performance on a certified reference material of the appropriate matrix and within the appropriate calibration range for the application. This is considered the best estimate of the true method sensitivity as opposed to a statistical determination based on the standard deviation of

replicate analyses of a low-concentration sample. While the statistical approach demonstrates the potential data variability for a given sample matrix at one point in time, it does not represent what can be detected or most importantly the lowest concentration that can be calibrated. For this reason the sensitivity should be established as the lowest point of detection based on acceptable target analyte recovery in the desired sample matrix.

9.7 Confirmatory samples -- The comparability of the FPXRF analysis is determined by submitting FPXRF-analyzed samples for analysis at a laboratory. The method of confirmatory analysis must meet the project and XRF measurement data quality objectives. The confirmatory samples must be splits of the well homogenized sample material. In some cases the prepared sample cups can be submitted. A minimum of 1 sample for each 20 FPXRF-analyzed samples should be submitted for confirmatory analysis. This frequency will depend on project-specific data quality objectives. The confirmatory analyses can also be used to verify the quality of the FPXRF data. The confirmatory samples should be selected from the lower, middle, and upper range of concentrations measured by the FPXRF. They should also include samples with analyte concentrations at or near the site action levels. The results of the confirmatory analysis and FPXRF analyses should be evaluated with a least squares linear regression analysis. If the measured concentrations span more than one order of magnitude, the data should be log-transformed to standardize variance which is proportional to the magnitude of measurement. The correlation coefficient (r) for the results should be 0.7 or greater for the FPXRF data to be considered screening level data. If the r is 0.9 or greater and inferential statistics indicate the FPXRF data and the confirmatory data are statistically equivalent at a 99 percent confidence level, the data could potentially meet definitive level data criteria.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument calibration -- Instrument calibration procedures vary among FPXRF instruments. Users of this method should follow the calibration procedures outlined in the operator's manual for each specific FPXRF instrument. Generally, however, three types of calibration procedures exist for FPXRF instruments, namely: FP calibration, empirical calibration, and the Compton peak ratio or normalization method. These three types of calibration are discussed below.

10.2 Fundamental parameters calibration -- FP calibration procedures are extremely variable. An FP calibration provides the analyst with a "standardless" calibration. The advantages of FP calibrations over empirical calibrations include the following:

- No previously collected site-specific samples are necessary, although site-specific samples with confirmed and validated analytical results for all elements present could be used.
- Cost is reduced because fewer confirmatory laboratory results or calibration standards are necessary.

However, the analyst should be aware of the limitations imposed on FP calibration by particle size and matrix effects. These limitations can be minimized by adhering to the preparation procedure described in Sec. 7.3. The two FP calibration processes discussed below are based on an effective energy FP routine and a back scatter with FP (BFP) routine. Each FPXRF FP calibration process is based on a different iterative algorithmic method. The calibration procedure for each routine is explained in detail in the manufacturer's user manual for each FPXRF instrument; in addition, training courses are offered for each instrument.

10.2.1 Effective energy FP calibration -- The effective energy FP calibration is performed by the manufacturer before an instrument is sent to the analyst. Although SSCS can be used, the calibration relies on pure element standards or SRMs such as those obtained from NIST for the FP calibration. The effective energy routine relies on the spectrometer response to pure elements and FP iterative algorithms to compensate for various matrix effects.

Alpha coefficients are calculated using a variation of the Sherman equation, which calculates theoretical intensities from the measurement of pure element samples. These coefficients indicate the quantitative effect of each matrix element on an analyte's measured x-ray intensity. Next, the Lachance Traill algorithm is solved as a set of simultaneous equations based on the theoretical intensities. The alpha coefficients are then downloaded into the specific instrument.

The working effective energy FP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of sampling. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. A manufacturer-recommended count time per source should be used for the calibration check. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A percent difference (%D) is then calculated for each target analyte. The %D should be within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line or the y-intercept value for the analyte. The SRM or SSCS is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

The equation to calibrate %D is as follows:

$$\%D = ((C_s - C_k) / C_k) \times 100$$

where:

%D = Percent difference
C_k = Certified concentration of standard sample
C_s = Measured concentration of standard sample

10.2.2 BFP calibration -- BFP calibration relies on the ability of the liquid nitrogen-cooled, Si(Li) solid-state detector to separate the coherent (Compton) and incoherent (Rayleigh) backscatter peaks of primary radiation. These peak intensities are known to be a function of sample composition, and the ratio of the Compton to Rayleigh peak is a function of the mass absorption of the sample. The calibration procedure is explained in detail in the instrument manufacturer's manual. Following is a general description of the BFP calibration procedure.

The concentrations of all detected and quantified elements are entered into the computer software system. Certified element results for an NIST SRM or confirmed and validated results for an SSCS can be used. In addition, the concentrations of oxygen and silicon must be entered; these two concentrations are not found in standard metals analyses. The manufacturer provides silicon and oxygen concentrations for typical soil types. Pure element standards are then analyzed using a manufacturer-recommended

count time per source. The results are used to calculate correction factors in order to adjust for spectrum overlap of elements.

The working BFP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of the analysis. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. The standard sample is analyzed using a manufacturer-recommended count time per source to check the calibration curve. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A %D is then calculated for each target analyte. The %D should fall within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line the y-intercept value for the analyte. The standard sample is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

10.3 Empirical calibration -- An empirical calibration can be performed with SSCS, site-typical standards, or standards prepared from metal oxides. A discussion of SSCS is included in Sec. 7.3; if no previously characterized samples exist for a specific site, site-typical standards can be used. Site-typical standards may be selected from commercially available characterized soils or from SSCS prepared for another site. The site-typical standards should closely approximate the site's soil matrix with respect to particle size distribution, mineralogy, and contaminant analytes. If neither SSCS nor site-typical standards are available, it is possible to make gravimetric standards by adding metal oxides to a "clean" sand or silicon dioxide matrix that simulates soil. Metal oxides can be purchased from various chemical vendors. If standards are made on site, a balance capable of weighing items to at least two decimal places is necessary. Concentrated ICP or AA standard solutions can also be used to make standards. These solutions are available in concentrations of 10,000 parts per million, thus only small volumes have to be added to the soil.

An empirical calibration using SSCS involves analysis of SSCS by the FPXRF instrument and by a conventional analytical method such as ICP or AA. A total acid digestion procedure should be used by the laboratory for sample preparation. Generally, a minimum of 10 and a maximum of 30 well characterized SSCS, site-typical standards, or prepared metal oxide standards are necessary to perform an adequate empirical calibration. The exact number of standards depends on the number of analytes of interest and interfering elements. Theoretically, an empirical calibration with SSCS should provide the most accurate data for a site because the calibration compensates for site-specific matrix effects.

The first step in an empirical calibration is to analyze the pure element standards for the elements of interest. This enables the instrument to set channel limits for each element for spectral deconvolution. Next the SSCS, site-typical standards, or prepared metal oxide standards are analyzed using a count time of 200 seconds per source or a count time recommended by the manufacturer. This will produce a spectrum and net intensity of each analyte in each standard. The analyte concentrations for each standard are then entered into the instrument software; these concentrations are those obtained from the laboratory, the certified results, or the gravimetrically determined concentrations of the prepared standards. This gives the instrument analyte values to regress against corresponding intensities during the modeling stage. The regression equation correlates the concentrations of an analyte with its net intensity.

The calibration equation is developed using a least squares fit regression analysis. After the regression terms to be used in the equation are defined, a mathematical equation can be developed to calculate the analyte concentration in an unknown sample. In some FPXRF instruments, the software of the instrument calculates the regression equation. The software uses calculated intercept and slope values to form a multiterm equation. In conjunction with the software in the instrument, the operator can adjust the multiterm equation to minimize interelement interferences and optimize the intensity calibration curve.

It is possible to define up to six linear or nonlinear terms in the regression equation. Terms can be added and deleted to optimize the equation. The goal is to produce an equation with the smallest regression error and the highest correlation coefficient. These values are automatically computed by the software as the regression terms are added, deleted, or modified. It is also possible to delete data points from the regression line if these points are significant outliers or if they are heavily weighing the data. Once the regression equation has been selected for an analyte, the equation can be entered into the software for quantitation of analytes in subsequent samples. For an empirical calibration to be acceptable, the regression equation for a specific analyte should have a correlation coefficient of 0.98 or greater or meet the DQOs of the project.

In an empirical calibration, one must apply the DQOs of the project and ascertain critical or action levels for the analytes of interest. It is within these concentration ranges or around these action levels that the FPXRF instrument should be calibrated most accurately. It may not be possible to develop a good regression equation over several orders of analyte concentration.

10.4 Compton normalization method -- The Compton normalization method is based on analysis of a single, certified standard and normalization for the Compton peak. The Compton peak is produced from incoherent backscattering of x-ray radiation from the excitation source and is present in the spectrum of every sample. The Compton peak intensity changes with differing matrices. Generally, matrices dominated by lighter elements produce a larger Compton peak, and those dominated by heavier elements produce a smaller Compton peak. Normalizing to the Compton peak can reduce problems with varying matrix effects among samples. Compton normalization is similar to the use of internal standards in organics analysis. The Compton normalization method may not be effective when analyte concentrations exceed a few percent.

The certified standard used for this type of calibration could be an NIST SRM such as 2710 or 2711. The SRM must be a matrix similar to the samples and must contain the analytes of interests at concentrations near those expected in the samples. First, a response factor has to be determined for each analyte. This factor is calculated by dividing the net peak intensity by the analyte concentration. The net peak intensity is gross intensity corrected for baseline reading. Concentrations of analytes in samples are then determined by multiplying the baseline corrected analyte signal intensity by the normalization factor and by the response factor. The normalization factor is the quotient of the baseline corrected Compton K_{α} peak intensity of the SRM divided by that of the samples. Depending on the FPXRF instrument used, these calculations may be done manually or by the instrument software.

11.0 PROCEDURE

11.1 Operation of the various FPXRF instruments will vary according to the manufacturers' protocols. Before operating any FPXRF instrument, one should consult the manufacturer's manual. Most manufacturers recommend that their instruments be allowed to warm up for 15 to 30 minutes before analysis of samples. This will help alleviate drift or energy calibration problems later during analysis.

11.2 Each FPXRF instrument should be operated according to the manufacturer's recommendations. There are two modes in which FPXRF instruments can be operated: in situ and intrusive. The in situ mode involves analysis of an undisturbed soil sediment or sample. Intrusive analysis involves collection and preparation of a soil or sediment sample before analysis. Some FPXRF instruments can operate in both modes of analysis, while others are designed to operate in only one mode. The two modes of analysis are discussed below.

11.3 For in situ analysis, remove any large or nonrepresentative debris from the soil surface before analysis. This debris includes rocks, pebbles, leaves, vegetation, roots, and concrete. Also, the soil surface must be as smooth as possible so that the probe window will have good contact with the surface. This may require some leveling of the surface with a stainless-steel trowel. During the study conducted to provide example performance data for this method, this modest amount of sample preparation was found to take less than 5 min per sample location. The last requirement is that the soil or sediment not be saturated with water. Manufacturers state that their FPXRF instruments will perform adequately for soils with moisture contents of 5 to 20 percent but will not perform well for saturated soils, especially if ponded water exists on the surface. Another recommended technique for in situ analysis is to tamp the soil to increase soil density and compactness for better repeatability and representativeness. This condition is especially important for heavy element analysis, such as barium. Source count times for in situ analysis usually range from 30 to 120 seconds, but source count times will vary among instruments and depending on the desired method sensitivity. Due to the heterogeneous nature of the soil sample, in situ analysis can provide only “screening” type data.

11.4 For intrusive analysis of surface or sediment, it is recommended that a sample be collected from a 4- by 4-inch square that is 1 inch deep. This will produce a soil sample of approximately 375 g or 250 cm^3, which is enough soil to fill an 8-ounce jar. However, the exact dimensions and sample depth should take into consideration the heterogeneous deposition of contaminants and will ultimately depend on the desired project-specific data quality objectives. The sample should be homogenized, dried, and ground before analysis. The sample can be homogenized before or after drying. The homogenization technique to be used after drying is discussed in Sec. 4.2. If the sample is homogenized before drying, it should be thoroughly mixed in a beaker or similar container, or if the sample is moist and has a high clay content, it can be kneaded in a plastic bag. One way to monitor homogenization when the sample is kneaded in a plastic bag is to add sodium fluorescein dye to the sample. After the moist sample has been homogenized, it is examined under an ultraviolet light to assess the distribution of sodium fluorescein throughout the sample. If the fluorescent dye is evenly distributed in the sample, homogenization is considered complete; if the dye is not evenly distributed, mixing should continue until the sample has been thoroughly homogenized. During the study conducted to provide data for this method, the time necessary for homogenization procedure using the fluorescein dye ranged from 3 to 5 min per sample. As demonstrated in Secs. 13.5 and 13.7, homogenization has the greatest impact on the reduction of sampling variability. It produces little or no contamination. Often, the direct analysis through the plastic bag is possible without the more labor intensive steps of drying, grinding, and sieving given in Secs. 11.5 and 11.6. Of course, to achieve the best data quality possible all four steps should be followed.

11.5 Once the soil or sediment sample has been homogenized, it should be dried. This can be accomplished with a toaster oven or convection oven. A small aliquot of the sample (20 to 50 g) is placed in a suitable container for drying. The sample should be dried for 2 to 4 hr in the convection or toaster oven at a temperature not greater than 150 EC. Samples may also be air dried under ambient temperature conditions using a 10- to 20-g portion. Regardless of what drying mechanism is used, the drying process is considered complete when a constant sample weight can be obtained. Care should be taken to avoid sample cross-contamination and these measures can be evaluated by including an appropriate method blank sample along with any sample preparation process.

CAUTION: Microwave drying is not a recommended procedure. Field studies have shown that microwave drying can increase variability between the FPXRF data and confirmatory analysis. High levels of metals in a sample can cause arcing in the microwave oven, and sometimes slag forms in the sample. Microwave oven drying can also melt plastic containers used to hold the sample.

11.6 The homogenized dried sample material should be ground with a mortar and pestle and passed through a 60-mesh sieve to achieve a uniform particle size. Sample grinding should continue until at least 90 percent of the original sample passes through the sieve. The grinding step normally takes an average of 10 min per sample. An aliquot of the sieved sample should then be placed in a 31.0-mm polyethylene sample cup (or equivalent) for analysis. The sample cup should be one-half to three-quarters full at a minimum. The sample cup should be covered with a 2.5 µm Mylar (or equivalent) film for analysis. The rest of the soil sample should be placed in a jar, labeled, and archived for possible confirmation analysis. All equipment including the mortar, pestle, and sieves must be thoroughly cleaned so that any cross-contamination is below the established lower limit of detection of the procedure or DQOs of the analysis. If all recommended sample preparation steps are followed, there is a high probability the desired laboratory data quality may be obtained.

12.0 DATA ANALYSIS AND CALCULATIONS

Most FPXRF instruments have software capable of storing all analytical results and spectra. The results are displayed in ppm and can be downloaded to a personal computer, which can be used to provide a hard copy printout. Individual measurements that are smaller than three times their associated SD should not be used for quantitation. See the manufacturer's instructions regarding data analysis and calculations.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 The sections to follow discuss three performance evaluation factors; namely, precision, accuracy, and comparability. The example data presented in Tables 4 through 8 were generated from results obtained from six FPXRF instruments (see Sec. 13.3). The soil samples analyzed by the six FPXRF instruments were collected from two sites in the United States. The soil samples contained several of the target analytes at concentrations ranging from "nondetect" to tens of thousands of mg/kg. These data are provided for guidance purposes only.

13.3 The six FPXRF instruments included the TN 9000 and TN Lead Analyzer manufactured by TN Spectrace; the X-MET 920 with a SiLi detector and X-MET 920 with a gas-filled proportional detector manufactured by Metorex, Inc.; the XL Spectrum Analyzer manufactured by Niton; and the MAP Spectrum Analyzer manufactured by Scitec. The TN 9000 and TN Lead Analyzer both have a HgI_2 detector. The TN 9000 utilized an Fe-55, Cd-109, and Am-241 source. The TN Lead Analyzer had only a Cd-109 source. The X-Met 920 with the SiLi detector had a Cd-109 and Am-241 source. The X-MET 920 with the gas-filled proportional detector had only a Cd-109 source. The XL Spectrum Analyzer utilized a silicon pin-diode

detector and a Cd-109 source. The MAP Spectrum Analyzer utilized a solid-state silicon detector and a Cd-109 source.

13.4 All example data presented in Tables 4 through 8 were generated using the following calibrations and source count times. The TN 9000 and TN Lead Analyzer were calibrated using fundamental parameters using NIST SRM 2710 as a calibration check sample. The TN 9000 was operated using 100, 60, and 60 second count times for the Cd-109, Fe-55, and Am-241 sources, respectively. The TN Lead analyzer was operated using a 60 second count time for the Cd-109 source. The X-MET 920 with the Si(Li) detector was calibrated using fundamental parameters and one well characterized site-specific soil standard as a calibration check. It used 140 and 100 second count times for the Cd-109 and Am-241 sources, respectively. The X-MET 920 with the gas-filled proportional detector was calibrated empirically using between 10 and 20 well characterized site-specific soil standards. It used 120 second times for the Cd-109 source. The XL Spectrum Analyzer utilized NIST SRM 2710 for calibration and the Compton peak normalization procedure for quantitation based on 60 second count times for the Cd-109 source. The MAP Spectrum Analyzer was internally calibrated by the manufacturer. The calibration was checked using a well-characterized site-specific soil standard. It used 240 second times for the Cd-109 source.

13.5 Precision measurements -- The example precision data are presented in Table 4. These data are provided for guidance purposes only. Each of the six FPXRF instruments performed 10 replicate measurements on 12 soil samples that had analyte concentrations ranging from "nondetects" to thousands of mg/kg. Each of the 12 soil samples underwent 4 different preparation techniques from in situ (no preparation) to dried and ground in a sample cup. Therefore, there were 48 precision data points for five of the instruments and 24 precision points for the MAP Spectrum Analyzer. The replicate measurements were taken using the source count times discussed at the beginning of this section.

For each detectable analyte in each precision sample a mean concentration, standard deviation, and RSD was calculated for each analyte. The data presented in Table 4 is an average RSD for the precision samples that had analyte concentrations at 5 to 10 times the lower limit of detection for that analyte for each instrument. Some analytes such as mercury, selenium, silver, and thorium were not detected in any of the precision samples so these analytes are not listed in Table 4. Some analytes such as cadmium, nickel, and tin were only detected at concentrations near the lower limit of detection so that an RSD value calculated at 5 to 10 times this limit was not possible.

One FPXRF instrument collected replicate measurements on an additional nine soil samples to provide a better assessment of the effect of sample preparation on precision. Table 5 shows these results. These data are provided for guidance purposes only. The additional nine soil samples were comprised of three from each texture and had analyte concentrations ranging from near the lower limit of detection for the FPXRF analyzer to thousands of mg/kg. The FPXRF analyzer only collected replicate measurements from three of the preparation methods; no measurements were collected from the in situ homogenized samples. The FPXRF analyzer conducted five replicate measurements of the in situ field samples by taking measurements at five different points within the 4-inch by 4-inch sample square. Ten replicate measurements were collected for both the intrusive undried and unground and intrusive dried and ground samples contained in cups. The cups were shaken between each replicate measurement.

Table 5 shows that the precision dramatically improved from the in situ to the intrusive measurements. In general there was a slight improvement in precision when the sample was dried and ground. Two factors caused the precision for the in situ measurements to be poorer. The major factor is soil heterogeneity. By moving the probe within the 4-inch by 4-inch square,

measurements of different soil samples were actually taking place within the square. Table 5 illustrates the dominant effect of soil heterogeneity. It overwhelmed instrument precision when the FPXRF analyzer was used in this mode. The second factor that caused the RSD values to be higher for the in situ measurements is the fact that only five instead of ten replicates were taken. A lesser number of measurements caused the standard deviation to be larger which in turn elevated the RSD values.

13.6 Accuracy measurements -- Five of the FPXRF instruments (not including the MAP Spectrum Analyzer) analyzed 18 SRMs using the source count times and calibration methods given at the beginning of this section. The 18 SRMs included 9 soil SRMs, 4 stream or river sediment SRMs, 2 sludge SRMs, and 3 ash SRMs. Each of the SRMs contained known concentrations of certain target analytes. A percent recovery was calculated for each analyte in each SRM for each FPXRF instrument. Table 6 presents a summary of this data. With the exception of cadmium, chromium, and nickel, the values presented in Table 6 were generated from the 13 soil and sediment SRMs only. The 2 sludge and 3 ash SRMs were included for cadmium, chromium, and nickel because of the low or nondetectable concentrations of these three analytes in the soil and sediment SRMs.

Only 12 analytes are presented in Table 6. These are the analytes that are of environmental concern and provided a significant number of detections in the SRMs for an accuracy assessment. No data is presented for the X-MET 920 with the gas-filled proportional detector. This FPXRF instrument was calibrated empirically using site-specific soil samples. The percent recovery values from this instrument were very sporadic and the data did not lend itself to presentation in Table 6.

Table 7 provides a more detailed summary of accuracy data for one particular FPXRF instrument (TN 9000) for the 9 soil SRMs and 4 sediment SRMs. These data are provided for guidance purposes only. Table 7 shows the certified value, measured value, and percent recovery for five analytes. These analytes were chosen because they are of environmental concern and were most prevalently certified for in the SRM and detected by the FPXRF instrument. The first nine SRMs are soil and the last 4 SRMs are sediment. Percent recoveries for the four NIST SRMs were often between 90 and 110 percent for all analytes.

13.7 Comparability -- Comparability refers to the confidence with which one data set can be compared to another. In this case, FPXRF data generated from a large study of six FPXRF instruments was compared to SW-846 Methods 3050 and 6010 which are the standard soil extraction for metals and analysis by inductively coupled plasma. An evaluation of comparability was conducted by using linear regression analysis. Three factors were determined using the linear regression. These factors were the y-intercept, the slope of the line, and the coefficient of determination (r^2).

As part of the comparability assessment, the effects of soil type and preparation methods were studied. Three soil types (textures) and four preparation methods were examined during the study. The preparation methods evaluated the cumulative effect of particle size, moisture, and homogenization on comparability. Due to the large volume of data produced during this study, linear regression data for six analytes from only one FPXRF instrument is presented in Table 8. Similar trends in the data were seen for all instruments. These data are provided for guidance purposes only.

Table 8 shows the regression parameters for the whole data set, broken out by soil type, and by preparation method. These data are provided for guidance purposes only. The soil types are as follows: soil 1--sand; soil 2--loam; and soil 3--silty clay. The preparation methods are as follows: preparation 1--in situ in the field; preparation 2--intrusive, sample collected and homogenized; preparation 3--intrusive, with sample in a sample cup but sample still wet and not

ground; and preparation 4–intrusive, with sample dried, ground, passed through a 40-mesh sieve, and placed in sample cup.

For arsenic, copper, lead, and zinc, the comparability to the confirmatory laboratory was excellent with r^2 values ranging from 0.80 to 0.99 for all six FPXRF instruments. The slopes of the regression lines for arsenic, copper, lead, and zinc, were generally between 0.90 and 1.00 indicating the data would need to be corrected very little or not at all to match the confirmatory laboratory data. The r^2 values and slopes of the regression lines for barium and chromium were not as good as for the other for analytes, indicating the data would have to be corrected to match the confirmatory laboratory.

Table 8 demonstrates that there was little effect of soil type on the regression parameters for any of the six analytes. The only exceptions were for barium in soil 1 and copper in soil 3. In both of these cases, however, it is actually a concentration effect and not a soil effect causing the poorer comparability. All barium and copper concentrations in soil 1 and 3, respectively, were less than 350 mg/kg.

Table 8 shows there was a preparation effect on the regression parameters for all six analytes. With the exception of chromium, the regression parameters were primarily improved going from preparation 1 to preparation 2. In this step, the sample was removed from the soil surface, all large debris was removed, and the sample was thoroughly homogenized. The additional two preparation methods did little to improve the regression parameters. This data indicates that homogenization is the most critical factor when comparing the results. It is essential that the sample sent to the confirmatory laboratory match the FPXRF sample as closely as possible.

Sec. 11.0 of this method discusses the time necessary for each of the sample preparation techniques. Based on the data quality objectives for the project, an analyst must decide if it is worth the extra time necessary to dry and grind the sample for small improvements in comparability. Homogenization requires 3 to 5 min. Drying the sample requires one to two hours. Grinding and sieving requires another 10 to 15 min per sample. Lastly, when grinding and sieving is conducted, time has to be allotted to decontaminate the mortars, pestles, and sieves. Drying and grinding the samples and decontamination procedures will often dictate that an extra person be on site so that the analyst can keep up with the sample collection crew. The cost of requiring an extra person on site to prepare samples must be balanced with the gain in data quality and sample throughput.

13.8 The following documents may provide additional guidance and insight on this method and technique:

13.8.1 A. D. Hewitt, "Screening for Metals by X-ray Fluorescence Spectrometry/Response Factor/Compton K_α Peak Normalization Analysis," American Environmental Laboratory, pp 24-32, 1994.

13.8.2 S. Piorek and J. R. Pasmore, "Standardless, In Situ Analysis of Metallic Contaminants in the Natural Environment With a PC-Based, High Resolution Portable X-Ray Analyzer," Third International Symposium on Field Screening Methods for Hazardous Waste and Toxic Chemicals, Las Vegas, Nevada, February 24-26, 1993, Vol 2, pp 1135-1151, 1993.

13.8.3 S. Shefsky, "Sample Handling Strategies for Accurate Lead-in-soil Measurements in the Field and Laboratory," *International Symposium of Field Screening Methods for Hazardous Waste and Toxic Chemicals*, Las Vegas, NV, January 29-31, 1997.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, http://www.acs.org.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. Metorex, X-MET 920 User's Manual.

2. Spectrace Instruments, "Energy Dispersive X-ray Fluorescence Spectrometry: An Introduction," 1994.

3. TN Spectrace, Spectrace 9000 Field Portable/Benchtop XRF Training and Applications Manual.

4. Unpublished SITE data, received from PRC Environment Management, Inc.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables referenced by this method. A flow diagram of the procedure follows the tables.

TABLE 1

EXAMPLE INTERFERENCE FREE LOWER LIMITS OF DETECTION

Analyte	Chemical Abstract Series Number	Lower Limit of Detection in Quartz Sand (milligrams per kilogram)
Antimony (Sb)	7440-36-0	40
Arsenic (As)	7440-38-0	40
Barium (Ba)	7440-39-3	20
Cadmium (Cd)	7440-43-9	100
Calcium (Ca)	7440-70-2	70
Chromium (Cr)	7440-47-3	150
Cobalt (Co)	7440-48-4	60
Copper (Cu)	7440-50-8	50
Iron (Fe)	7439-89-6	60
Lead (Pb)	7439-92-1	20
Manganese (Mn)	7439-96-5	70
Mercury (Hg)	7439-97-6	30
Molybdenum (Mo)	7439-93-7	10
Nickel (Ni)	7440-02-0	50
Potassium (K)	7440-09-7	200
Rubidium (Rb)	7440-17-7	10
Selenium (Se)	7782-49-2	40
Silver (Ag)	7440-22-4	70
Strontium (Sr)	7440-24-6	10
Thallium (Tl)	7440-28-0	20
Thorium (Th)	7440-29-1	10
Tin (Sn)	7440-31-5	60
Titanium (Ti)	7440-32-6	50
Vanadium (V)	7440-62-2	50
Zinc (Zn)	7440-66-6	50
Zirconium (Zr)	7440-67-7	10

Source: Refs. 1, 2, and 3
These data are provided for guidance purposes only.

TABLE 2

SUMMARY OF RADIOISOTOPE SOURCE CHARACTERISTICS

Source	Activity (mCi)	Half-Life (Years)	Excitation Energy (keV)	Elemental Analysis Range	
Fe-55	20-50	2.7	5.9	Sulfur to Chromium Molybdenum to Barium	K Lines L Lines
Cd-109	5-30	1.3	22.1 and 87.9	Calcium to Rhodium Tantalum to Lead Barium to Uranium	K Lines K Lines L Lines
Am-241	5-30	432	26.4 and 59.6	Copper to Thulium Tungsten to Uranium	K Lines L Lines
Cm-244	60-100	17.8	14.2	Titanium to Selenium Lanthanum to Lead	K Lines L Lines

Source: Refs. 1, 2, and 3

TABLE 3

SUMMARY OF X-RAY TUBE SOURCE CHARACTERISTICS

Anode Material	Recommended Voltage Range (kV)	K-alpha Emission (keV)	Elemental Analysis Range	
Cu	18-22	8.04	Potassium to Cobalt Silver to Gadolinium	K Lines L Lines
Mo	40-50	17.4	Cobalt to Yttrium Europium to Radon	K Lines L Lines
Ag	50-65	22.1	Zinc to Technicium Ytterbium to Neptunium	K Lines L Lines

Source: Ref. 4

Notes: The sample elements excited are chosen by taking as the lower limit the same ratio of excitation line energy to element absorption edge as in Table 2 (approximately 0.45) and the requirement that the excitation line energy be above the element absorption edge as the upper limit (L2 edges used for L lines). K-beta excitation lines were ignored.

TABLE 4

EXAMPLE PRECISION VALUES

Analyte	Average Relative Standard Deviation for Each Instrument at 5 to 10 Times the Lower Limit of Detection					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	6.54	NR	NR	NR	NR	NR
Arsenic	5.33	4.11	3.23	1.91	12.47	6.68
Barium	4.02	NR	3.31	5.91	NR	NR
Cadmium	29.84[a]	NR	24.80[a]	NR	NR	NR
Calcium	2.16	NR	NR	NR	NR	NR
Chromium	22.25	25.78	22.72	3.91	30.25	NR
Cobalt	33.90	NR	NR	NR	NR	NR
Copper	7.03	9.11	8.49	9.12	12.77	14.86
Iron	1.78	1.67	1.55	NR	2.30	NR
Lead	6.45	5.93	5.05	7.56	6.97	12.16
Manganese	27.04	24.75	NR	NR	NR	NR
Molybdenum	6.95	NR	NR	NR	12.60	NR
Nickel	30.85[a]	NR	24.92[a]	20.92[a]	NA	NR
Potassium	3.90	NR	NR	NR	NR	NR
Rubidium	13.06	NR	NR	NR	32.69[a]	NR
Strontium	4.28	NR	NR	NR	8.86	NR
Tin	24.32[a]	NR	NR	NR	NR	NR
Titanium	4.87	NR	NR	NR	NR	NR
Zinc	7.27	7.48	4.26	2.28	10.95	0.83
Zirconium	3.58	NR	NR	NR	6.49	NR

These data are provided for guidance purposes only.
Source: Ref. 4

[a] These values are biased high because the concentration of these analytes in the soil samples was near the lower limit of detection for that particular FPXRF instrument.

NR Not reported.

NA Not applicable; analyte was reported but was below the established lower limit detection.

TABLE 5

EXAMPLES OF PRECISION AS AFFECTED BY SAMPLE PREPARATION

Analyte	Average Relative Standard Deviation for Each Preparation Method		
	In Situ-Field	Intrusive-Undried and Unground	Intrusive-Dried and Ground
Antimony	30.1	15.0	14.4
Arsenic	22.5	5.36	3.76
Barium	17.3	3.38	2.90
Cadmium[a]	41.2	30.8	28.3
Calcium	17.5	1.68	1.24
Chromium	17.6	28.5	21.9
Cobalt	28.4	31.1	28.4
Copper	26.4	10.2	7.90
Iron	10.3	1.67	1.57
Lead	25.1	8.55	6.03
Manganese	40.5	12.3	13.0
Mercury	ND	ND	ND
Molybdenum	21.6	20.1	19.2
Nickel[a]	29.8	20.4	18.2
Potassium	18.6	3.04	2.57
Rubidium	29.8	16.2	18.9
Selenium	ND	20.2	19.5
Silver[a]	31.9	31.0	29.2
Strontium	15.2	3.38	3.98
Thallium	39.0	16.0	19.5
Thorium	NR	NR	NR
Tin	ND	14.1	15.3
Titanium	13.3	4.15	3.74
Vanadium	NR	NR	NR
Zinc	26.6	13.3	11.1
Zirconium	20.2	5.63	5.18

These data are provided for guidance purposes only.
Source: Ref. 4
[a] These values may be biased high because the concentration of these analytes in the soil samples was near the lower limit of detection.
ND Not detected.
NR Not reported.

TABLE 6

EXAMPLE ACCURACY VALUES

Analyte	Instrument															
	TN 9000				TN Lead Analyzer				X-MET 920 (SiLi Detector)				XL Spectrum Analyzer			
	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec	SD	n	Range of % Rec.	Mean % Rec.	SD
Sb	2	100-149	124.3	NA	--	--	--	--	--	--	--	--	--	--	--	--
As	5	68-115	92.8	17.3	5	44-105	83.4	23.2	4	9.7-91	47.7	39.7	5	38-535	189.8	206
Ba	9	98-198	135.3	36.9	--	--	--	--	9	18-848	168.2	262	--	--	--	--
Cd	2	99-129	114.3	NA	--	--	--	--	6	81-202	110.5	45.7	--	--	--	--
Cr	2	99-178	138.4	NA	--	--	--	--	7	22-273	143.1	93.8	3	98-625	279.2	300
Cu	8	61-140	95.0	28.8	6	38-107	79.1	27.0	11	10-210	111.8	72.1	8	95-480	203.0	147
Fe	6	78-155	103.7	26.1	6	89-159	102.3	28.6	6	48-94	80.4	16.2	6	26-187	108.6	52.9
Pb	11	66-138	98.9	19.2	11	68-131	97.4	18.4	12	23-94	72.7	20.9	13	80-234	107.3	39.9
Mn	4	81-104	93.1	9.70	3	92-152	113.1	33.8	--	--	--	--	--	--	--	--
Ni	3	99-122	109.8	12.0	--	--	--	--	--	--	--	--	3	57-123	87.5	33.5
Sr	8	110-178	132.6	23.8	--	--	--	--	--	--	--	--	7	86-209	125.1	39.5
Zn	11	41-130	94.3	24.0	10	81-133	100.0	19.7	12	46-181	106.6	34.7	11	31-199	94.6	42.5

Source: Ref. 4. These data are provided for guidance purposes only.
n: Number of samples that contained a certified value for the analyte and produced a detectable concentration from the FPXRF instrument.
SD: Standard deviation; NA: Not applicable; only two data points, therefore, a SD was not calculated.
%Rec.: Percent recovery.
-- No data.

TABLE 7

EXAMPLE ACCURACY FOR TN 9000[a]

Standard Reference Material	Arsenic			Barium			Copper			Lead			Zinc		
	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.
RTC CRM-021	24.8	ND	NA	586	1135	193.5	4792	2908	60.7	144742	149947	103.6	546	224	40.9
RTC CRM-020	397	429	92.5	22.3	ND	NA	753	583	77.4	5195	3444	66.3	3022	3916	129.6
BCR CRM 143R	--	--	--	--	--	--	131	105	80.5	180	206	114.8	1055	1043	99.0
BCR CRM 141	--	--	--	--	--	--	32.6	ND	NA	29.4	ND	NA	81.3	ND	NA
USGS GXR-2	25.0	ND	NA	2240	2946	131.5	76.0	106	140.2	690	742	107.6	530	596	112.4
USGS GXR-6	330	294	88.9	1300	2581	198.5	66.0	ND	NA	101	80.9	80.1	118	ND	NA
NIST 2711	105	104	99.3	726	801	110.3	114	ND	NA	1162	1172	100.9	350	333	94.9
NIST 2710	626	722	115.4	707	782	110.6	2950	2834	96.1	5532	5420	98.0	6952	6476	93.2
NIST 2709	17.7	ND	NA	968	950	98.1	34.6	ND	NA	18.9	ND	NA	106	98.5	93.0
NIST 2704	23.4	ND	NA	414	443	107.0	98.6	105	106.2	161	167	103.5	438	427	97.4
CNRC PACS-1	211	143	67.7	--	772	NA	452	302	66.9	404	332	82.3	824	611	74.2
SARM-51	--	--	--	335	466	139.1	268	373	139.2	5200	7199	138.4	2200	2676	121.6
SARM-52	--	--	--	410	527	128.5	219	193	88.1	1200	1107	92.2	264	215	81.4

Source: Ref. 4. These data are provided for guidance purposes only.
[a] All concentrations in milligrams per kilogram.
%Rec.: Percent recovery; ND: Not detected; NA: Not applicable.
-- No data.

TABLE 8

EXAMPLE REGRESSION PARAMETERS FOR COMPARABILITY[1]

	Arsenic				Barium				Copper			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	824	0.94	1.62	0.94	1255	0.71	60.3	0.54	984	0.93	2.19	0.93
Soil 1	368	0.96	1.41	0.95	393	0.05	42.6	0.11	385	0.94	1.26	0.99
Soil 2	453	0.94	1.51	0.96	462	0.56	30.2	0.66	463	0.92	2.09	0.95
Soil 3	—	—	—	—	400	0.85	44.7	0.59	136	0.46	16.60	0.57
Prep 1	207	0.87	2.69	0.85	312	0.64	53.7	0.55	256	0.87	3.89	0.87
Prep 2	208	0.97	1.38	0.95	315	0.67	64.6	0.52	246	0.96	2.04	0.93
Prep 3	204	0.96	1.20	0.99	315	0.78	64.6	0.53	236	0.97	1.45	0.99
Prep 4	205	0.96	1.45	0.98	313	0.81	58.9	0.55	246	0.96	1.99	0.96

	Lead				Zinc				Chromium			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	1205	0.92	1.66	0.95	1103	0.89	1.86	0.95	280	0.70	64.6	0.42
Soil 1	357	0.94	1.41	0.96	329	0.93	1.78	0.93	—	—	—	—
Soil 2	451	0.93	1.62	0.97	423	0.85	2.57	0.90	—	—	—	—
Soil 3	397	0.90	2.40	0.90	351	0.90	1.70	0.98	186	0.66	38.9	0.50
Prep 1	305	0.80	2.88	0.86	286	0.79	3.16	0.87	105	0.80	66.1	0.43
Prep 2	298	0.97	1.41	0.96	272	0.95	1.86	0.93	77	0.51	81.3	0.36
Prep 3	302	0.98	1.26	0.99	274	0.93	1.32	1.00	49	0.73	53.7	0.45
Prep 4	300	0.96	1.38	1.00	271	0.94	1.41	1.01	49	0.75	31.6	0.56

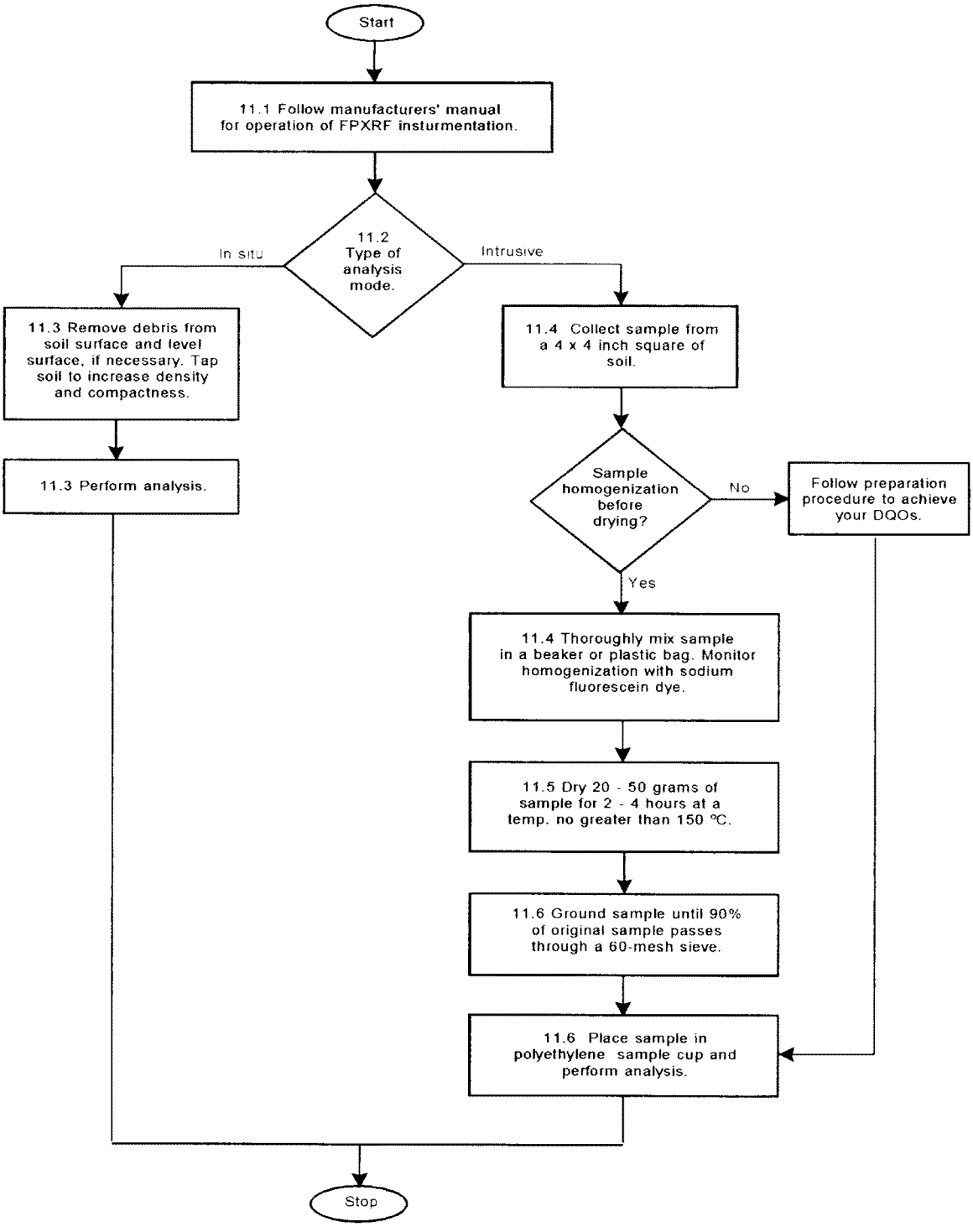
Source: Ref. 4. These data are provided for guidance purposes only.

[1] Log-transformed data

n: Number of data points; r^2: Coefficient of determination; Int.: Y-intercept

— No applicable data

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT



APPENDIX C
SAMPLE XRF DATA SHEET

PARSONS

LBP EXTERIOR XRF ANALYSES

				XRF Serial No.:	
Log Date:		Project No:	449646	Property No.:	
Inspector:		Project Name:	DTSC Exide Off-Site	Property Address:	

SAMPLE ID	COMPOSITION	SUBSTRATE	COLOR	SIDE	CONC.	+/-	UNITS	COMMENTS

PARSONS

Soil Sample XRF Analyses

Project Name: DTSC Exide Off-Site
Project Number: 449646
Log Date: ____________________
Inspector: ____________________
XRF Serial No.: ____________________
Property No.: ____________________
Property Address: ____________________

SAMPLE ID	DEPTH INTERVAL	LEAD CONC.	+/-	UNITS	COMMENTS